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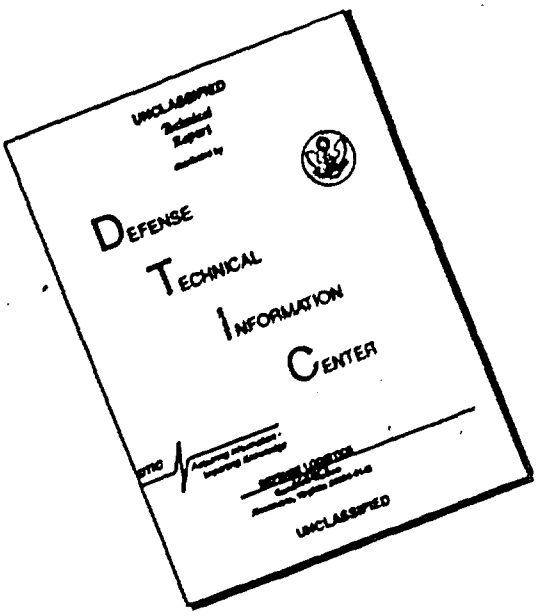

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AZUSA PLANT

STRUCTURAL MATERIALS DIVISION

DEVELOPMENT OF IMPROVED HIGH-STRENGTH PREIMPREGNATED MATERIALS FOR FILAMENT-WINDING

A REPORT TO

BUREAU OF NAVAL WEAPONS

CONTRACT NOW 61-0642-C (FBM)

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AEROJET GENERAL TIRE GENERAL

AEROJET-GENERAL CORPORATION

AZUSA, CALIFORNIA

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Report No. 2577 June 1963

AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

FOREWORD

This is a summary report of the activities of the Aerojet-General Corporation, covering the period of 26 June 1961 through 1 February 1963, on Contract Number NOw 61-0642-c(FBM). This contract is under the direct supervision of the Special Projects Office of the Bureau of Naval Weapons, with H. Bernstein acting as technical monitor.

This program is being conducted by the Materials Engineering Department of the Structural Materials Division, Aerojet-General Corporation, Azusa. Major responsibility for the program resides with Ira Petker. Other significant contributors to the program include Dr. S. Brelant and M. Segimoto.

AEROJET-GENERAL CORPORATION

Dr. S. Brelant, Head
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ABSTRACT

This program was a study of the processes used in the manufacture of 20-end ECG fibrous-glass roving and preimpregnated roving made from it. The program was composed of two independent phases. Phase I was a critical analysis of the various steps involved in the manufacture of glass roving from the point of drawing glass filaments to packaging and shipment of the product. The purpose was to establish optimum procedures which would lead to a retention of a greater proportion of the fiber's "virgin" strength. Test results for eight experimental lots of roving produced for this study are presented. Data includes gravimetric measurements and tensile strength of the AeroROVE strands, NOL rings, and 18-in.-dia chamber for both dry and preimpregnated rovings. Production preimpregnated rovings in which were incorporated improvements developed during this program have shown a tensile strength increase of approximately 25%. Also, improvements resulting in more uniform lineal weight and sizing content of roving have been achieved.

Phase II was a prepreg manufacturing process variable study. The purpose of this study was to obtain a clearer and more quantitative understanding of the effects of preimpregnation manufacturing parameters on the properties of prepreg roving. Sixteen experimental runs were made to investigate seven process variables, and two runs were made to improve and control resin content in prepregs by application of specially designed devices. Inspection test data for all these material lots are presented, as well as specific process conditions used to produce these lots. These data include gravimetric measurements (volatile, resin content, and weight per linear yard), resin flow, gel time, viscosity index, and tensile strength (both AeroROVE strands and NOL rings). A viscosity-index test was developed to measure the degree of resin polymerization and applied successfully to the analysis of the effects and the sensitivity of prepreg to variations in the parameters of preimpregnating processing. An approach to setting quantitative control limits in the manufacture of prepreg has been demonstrated by the application of the viscosity-index test as a means of correlating differences in processing characteristics of prepreg.

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I. INTRODUCTION

This program was a joint effort of Owens-Corning Fiberglas Corp., U.S. Polymeric Chemicals, Inc., and Aerojet-General Corp. The purpose of the program was to develop an improved, preimpregnated glass-fiber material (prepreg) suitable for use in filament-wound motor cases capable of withstanding tensile strength (fiber-stress) levels of from 375,000 to 400,000 psi.

The original program was to cover an 8-month period and was to be divided into two phases. Phase I was to establish optimum procedures for fabrication, handling, and shipping glass roving and for applying controlled quantities of resin to glass-roving materials. Specific tasks on Phase I were: (a) fabrication of eight 100-lb lots of E-glass with HTS sizing by Owens-Corning under controlled, documented conditions; (b) determination by Aerojet of the mechanical properties of each lot of glass; (c) impregnation by U.S. Polymeric of each lot of glass under controlled, documented conditions; (d) evaluation of each lot of prepreg by Aerojet; and (e) preparation of tentative material and process specifications for high-strength, high-quality prepreg. Phase II was to be a confirmation of the positive results of Phase I with a larger sampling of material prepared under production conditions.

Improvements in strength of production glass roving by incorporating changes which were developed for the first few lots of this program negated the need for Phase II as originally planned. Phase II was changed to a critical study of the preimpregnation process and its effect upon the processing characteristics of prepreg. Since Phases I and II in the revised program were essentially independent studies, for the sake of clarity they will be treated separately in this report.

II. PHASE I - STUDY OF ROVING FORMING PROCESS

A. DESCRIPTION OF STUDY

In filament winding, the basic type of reinforcement currently in use is unwoven glass filament in the form of multiple-end roving. Figure 1 is a schematic rendering of the process which is used to produce roving and includes all the basic operations applied to the glass fiber prior to its impregnation with a resin. The important steps in the process shown in Figure 1 are as follows:

1. Blending of raw materials used in the glass (A)
2. Melting of the glass raw materials (B)
3. Formation of the glass marbles (C)
4. Remelting of glass marbles in a bushing (D)
5. Gravity extrusion of glass through orifices in the bushing and formation of "virgin" fibers (E)
6. Application of size or finish to fibers (F)
7. Collection of fibers into a single end (G)
8. Winding of single end onto forming tube or cake package (H)
9. Oven bakeout of forming tube to remove excess volatiles from sizing (I)
10. Placement of a group of forming tubes on a creel (J)
11. Collection of a group of single ends through a guide eye to form a roving (K)
12. Windup of the roving to form a roving spool (L)
13. Packaging and shipment of roving (M).

Just after Step 5 (Figure 1,E) fibers formed from E-type glass composition have been reported by numerous observers to have strength in excess of 500,000 psi. A short time ago only 50% of this strength, or 250,000 psi, was retained when multiple end roving was tested after Step 13. The purpose of

II Phase I - Study of Roving Forming Process, A (cont.)

Phase I of this program was to analyze critically all steps in the roving forming process between Steps 5 and 13 for their contribution to this loss in strength. New methods were then to be developed and applied to those points in the process which would lead to a retention of a greater proportion of the original strength of the fibers.

The approach taken to this problem involved the preparation of experimental lots of roving by Owens-Corning. Each experimental lot incorporated certain potential improvements and consisted of ten 10-lb rolls of 20-end roving. Each lot was 100% inspected by Owens-Corning, using the following tests.*

lay	strand yardage
waywind	roving yardage
package length	strand moisture
diameter	strand volatiles
roving weight, net	strand solids
centering on tube	roving moisture
strand width	roving volatiles
strand spacing	roving solids
stops/doff	wetout rate
fuzz content	laminate clarity
stiffness	shelling
ribbonization	catenary
end count	durometer hardness
	tensile strength

Each experimental lot of roving was shipped to Aerojet, Structural Materials Division, where the following tests were performed: (1) vinyl strand strength, (2) 1/16-in. NOL-ring strength, (3) ignition loss, (4) weight per lineal yard, and (5) 18-in.-dia chamber strength.

The roving was then preimpregnated by U.S. Polymeric with E-787 type resin under constant impregnating conditions. It was then tested again by Aerojet for possible strength losses due to preimpregnation. The prepreg tests applied by Aerojet were the following: (1) prepreg strand strength, (2) 1/6-in. NOL-ring strength, (3) volatile content, (4) resin content, and (5) 18-in.-dia chamber strength.

*Owens-Corning Fiberglas Corp. Customer Acceptance Standards TP-245, 21 September 1961.

II Phase I - Study of Roving Forming Process, A (cont.)

All significant changes leading to improved strength or quality in the roving which were noted during the course of this program were to be incorporated into the standard production process by Owens-Corning.

B. DESCRIPTION OF EXPERIMENTAL LOTS OF ROVING

1. Lot 1 - Improved Package

a. More uniform tension was applied to each individual strand (end) through the use of more efficient guide eyes, alignment, and positioning. Automatic knock-off devices were maintained in the thread line to aid in assuring absolute end count (Step 11)*.

b. Incorporation of an "interlocking waywind." This refers to the internal characteristic of the roving package in the sense that each successive band (wrap) tends to lock preceding bands in place. Its use is to give greater stability to the roving package and to prevent sloughing (Step 12).

c. Development of a flatter band. This was accomplished through use of newer techniques in the areas of winding equipment, guides, and roving-package tensioning (Step 12).

d. Use of a stronger roving ball core. The need for greater strength was to preclude collapsing of the ball after preparation and during shipping (Steps 12 and 13).

e. End caps were employed to protect the prepared ball during handling, shipping, and storage. The core was changed for the same basic reason to a longer tube with a knurled surface to help prevent slipping of the ball on the core after production and to allow for end pads and caps (Step 13).

f. The roving was sealed in a polyethylene bag to protect it from contamination by moisture and foreign matter and to impress upon production personnel the "special" nature of the product being manufactured (Step 13).

g. The package hardness characteristics were improved by tightening the winding specifications to achieve more consistent measurements and

*Numbers in parentheses in Section II,B refer to the respective processing steps described in Section II,A.

II Phase I - Study of Roving Forming Process, B (cont.)

and setting minimum hardness limits in order to achieve better product uniformity (Step 12).

h. A larger and stronger shipping carton was developed to provide necessary space to package the roving, including changes made in d, e, and f above, and for additional protection to the product during shipping and warehousing (Step 13).

i. A new palletizing method was used consisting of three layers per pallet (24 cartons per layer) with corrugated layer caps over each layer to interlock the load. Damage-free freight cars were used to permit separation of a carload lot by means of cross bars in such a manner as to preclude shifting of the entire load in transit and to guard against other damage (Step 13).

2. Lot 2 - More Uniform Surface Treatment (Step 10)

a. Only a portion of each forming tube was used based on sizing migration curves. That portion where lowest variation was measured was used to prepare this lot of roving.

b. Distribution or migration curves of variation of sizing content within forming cake packages have been developed for HTS forming stock.* Various degrees of migration are measurable in many types of forming stock, including HTS.

3. Lot 3 - Improved Strand and Roving Weight Variation

a. Input was selected for uniformity in strand coating plus better-than-standard control of bare glass yards per pound (Steps 5 and 6).

b. Only two balls of roving were prepared from each set of 20 pre-tested tubes to minimize variation in weight per yard due to possible migration effects. This method was used in an attempt to asure a minimum variation in weight per yard throughout the sample roving packages.

*Additional details considered proprietary information by Owens-Corning and therefore not supplied by them.

II Phase I - Study of Roving Forming Process, B (cont.)

4. Lot 4 - Roving Stiffness Variation (Step 9)

Several methods of increasing the stiffness of HTS roving were investigated. The method which was selected yielded the most consistent results with a minimum difference in other characteristics or measurable properties. Additional heat application was the approach taken.*

5. Lot 5 - Package Moisture Variation

The roving was exposed to high humidity conditions for a period of time and rechecked for evidence of moisture penetration. Testing by Owens-Corning revealed moisture introduction at a level of 0.1% or less. Re-testing of significant properties exhibited lower degree of wetout. (The conclusion of Owens-Corning was that a meaningful amount of moisture had been introduced into interstices of the roving package. However, it was their contention that the strand coating had not been penetrated to any significant degree, if at all.)

6. Lot 6 - Catenary Improvement, Parallel-Wound Package

a. According to Owens-Corning, the best thinking of Owens-Corning and winding equipment manufacturers was combined in several attempts to reduce catenary, but they were unable to improve upon the available package supplied for Lot 1.

b. Pre-tested stock was prepared to be fabricated into roving by the Ashton Product Development Laboratory (OCF). After preparation four packages were sent to the Anderson plant for testing. Results did not indicate an improvement in catenary reduction. Laboratory personnel believe that the nature of a helical-wound package was the major reason for their failure to accomplish catenary reduction.

c. It was decided to use a level-wound package for this lot. The level-wound or parallel-wound package was, in the estimation of Aerojet and Owens-Corning, the best way to proceed. Essentially, parallel wraps were placed on a double-flanged spool with spacing between successive wraps. An

*Additional details considered proprietary information by Owens-Corning and therefore not supplied by them.

available smaller-capacity spool was employed (Step 12).

7. Lot 7 - Finer Filament Input Stock (Step 5)

DE filaments (approximately 408 filaments per end) were used in place of the standard G filaments (204 filaments per end).

8. Lot 8 - Zero Ribbonization

Methods employed were a combination of heat and chemical differences. The roving was otherwise made as nearly as possible in conformance with OCF CAS TP-245 (Step 9).

C. PREIMPREGNATION

In this program glass roving, after testing by Aerojet, was sent to U.S. Polymeric, where it was coated with E-787 type resin. Figure 2 is a schematic diagram of a typical impregnation process used to produce prepreg. The main steps in the preimpregnation process are as follows: (1) positioning of the roving spools on a creel, (2) coating of roving with resin by passing roving through a diluted solution of resin, (3) removal of solvent and partial polymerization of resin by means of heat within the drying tower, and (4) windup of B-staged prepreg.

For the preimpregnation of the experimental lots of roving, no deviations were allowed in the impregnation process except as required to control resin content. The important processing parameters and their levels were:

Parameter	Average Level
Tower temperature	360 ±10°F
Processing speed	61 ±10 ft/min
Creel tension	750 g
Resin-gel time	4 ±0.25 min

D. TEST RESULTS

Test data for all materials is summarized in Table 1 for dry glass and in Table 2 for prepreg. Data from 18-in. chambers is summarized in Table 3. As shown in Table 1, the average lot strength of vinyl-coated strands for all

lots was between 302,000 and 348,000 psi. Five of the eight lots, Lots 1, 3, 4, 5 and 7, had strand strength within a much narrower range, from 335,000 to 348,000 psi. The two lots with the lowest strength, about 303,000 psi, had low ribbonization. The average lot NOL-ring fiber strength was between 319,000 and 349,000 psi for the same five lots which had high strand strength. Lot 2 and the lots with low ribbonization (Lots 6 and 8) had NOL-ring fiber strengths of about 303,000 psi. Sizing content, as measured by ignition loss, tended to be quite consistent with a nominal average for all lots of about 1.45%. The weight per yard was also quite consistent at about 0.652 g.

As shown in Table 2, the lot average range for prepreg strands was between 292,000 and 340,000 psi. In general, the prepreg strand values were lower than the vinyl strand values for the same lots. However, NOL rings were very consistent between lots and also tended to be stronger than the equivalent in-process rings. With the exception of Lots 3 and 6, the lot average range for prepreg NOL rings was 335,000 to 352,000 psi. In most instances, the prepreg NOL rings failed at higher stress values than vinyl-coated strands from the same lot. A particularly large difference of this type was noted for Lot 8, in which the average of 304,000 psi for vinyl strands is considerably lower than the figure of 352,000 psi for prepreg NOL rings. The prepreg gravimetric data tends to vary somewhat more than is desirable, although in respect to resin content, Lot 7 (DE filaments) had an unusually small total variation between rolls (1%).

The data from the 18-in.-chamber tests, summarized in Table 3, does not indicate any consistent trend. Several chambers, particularly those prepared from Lots 3 and 5 prepreg, had hoop-filament stresses greater than 330,000 psi. However, other chambers had hoop filament stresses as low as 260,000 psi.

E. TECHNICAL DISCUSSION

1. Glass Strength

Certain inconsistencies appear in the test data when the data between dry glass and prepreg material, and from one test to another is compared. The prepreg strand strength is generally lower than the vinyl-coated strand strength, whereas the prepreg NOL-ring strength is higher than in-process NOL

II Phase I - Study of Roving Forming Process, E (cont.)

ring strength. It was also found that the vinyl strand strength was higher in general than in-process NOL-ring strength. However, prepreg strand is consistently lower than prepreg NOL ring strength. Finally, for certain lots, including Lots 2, 4, 5, 6, 7 and 8, the prepreg NOL-ring strength is equal to or higher than the vinyl strand strength measured for the same glass prior to preimpregnation. Since strand tests, NOL-ring tests, and 18-in.-chamber tests are the criteria used to evaluate improvements in the prepreg, a discussion and interpretation of this data at this point appears warranted.

The strand test is essentially a test of pure tension, whereas NOL ring testing, by its nature, imposes bending and shear forces which will cause glass failure at a lower stress level than the same glass fibers in pure tension. If the hypotheses are made that the glass fibers are strongest when stressed in pure tension, and that the preimpregnation process may or may not lower, but will not improve, the glass strength, the data can be interpreted more meaningfully. First, the highest glass strengths within any given lot of roving should have been for the vinyl strands; these values should be higher than the corresponding NOL-ring strengths. Since this was not true in several instances, it can be concluded that the vinyl strand test does not stress the glass to its ultimate capability; actual strength of the glass, therefore, is probably higher than is indicated by the test data, and the strand test data should be viewed as defining minimum rather than ultimate strength. The inability of the vinyl strand test to approach ultimate glass capabilities is associated with the properties of the vinyl resin and with the methods of gripping. A resin which has better wetting characteristics, higher shear strength, and more efficient shear transfer characteristics than the vinyl resin employed (i.e., the same type of resin used in chamber fabrication) would probably produce higher calculated glass stresses. Also, an improved method for distributing the load in the grip area more uniformly would also reduce borderline grip failures.*

The results obtained with prepreg strands are consistently lower than those obtained with prepreg NOL rings, indicating again that problems

*Recent work in the Structural Materials Division has tended to verify these conclusions. Further indications have been that all strand tests currently in use suffer from these same problems.

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associated with the test methods have introduced some complication to the simple analysis of the test data. However, the lower strength of the prepreg strand, compared to the vinyl strand, still requires an explanation, since prepreg NOL rings and 18-in.-chambers have consistently been equivalent to the higher end-strength and in-process NOL rings and chambers made with the same glass. It is particularly important to note that prepreg strand data is the result of a test of a composite rather than a simple test of glass strength. Although the amount of resin present in prepreg strands is theoretically great enough to completely impregnate the fibers, there is little excess resin available to make up for localized resin deficiencies, or to prevent sequential failure and peeling at localized fiber breaks. In addition to the low resin content, no pressure is used in preparing specimens; therefore, little effective resin migration takes place, which could compensate for local resin inadequacies and which could produce more uniform impregnation. Finally, in the grip area, little excess resin is available to protect the fibers and distribute the loads created by the friction grips. The sum total of these factors can make prepreg strands very sensitive to minor variations in resin distribution, spatial relations between ends, band width, position of filament flaws, etc. Indeed, roving impregnated in the laboratory, using the same resin systems, has yielded prepreg strand data which is equivalent to or higher than the vinyl strands.

Some preliminary work has been done with vinyl-coated prepreg strands, the results of which are shown in Table 4. All these strands were prepared in the same manner as standard prepreg strands, except that the prepreg was coated with vinyl resin prior to oven cure. In each case, the vinyl coating increased the failure stress significantly; the most dramatic effect was noted on the strands which orginally failed at the lower stress levels. This data indicates that improvements in strand grips which tend to distribute gripping loads more satisfactorily will lead to higher measured stress values.

Another interesting comparison was made between prepreg and in-process NOL rings; it was found that the prepreg rings were consistently higher in strength than in-process rings for the same lots of glass. Based on the original hypothesis that preimpregnation cannot improve glass strength, the

explanation for the strength improvement must be found in factors other than glass strength. Although no quantitative analysis is possible at this time, it would appear that the differences would be related to composite differences such as resin content, void content, interfacial shear, etc., or to resin differences such as shear strength, wettability, rigidity, etc. Since ring testing appears to be sensitive to one or more of these factors and does not measure a true physical property characteristic of any individual component of the composite, caution must be exercised in attributing differences in NOL-ring strength to differences in the glass strength.

2. Gravimetric Data

a. Roving

Ignition loss (or sizing content) for all experimental lots of material, except Lot 2, has been very consistent both between lots and between rolls within a given lot. (Lot 7 is an exception but this is probably because Lot 7 represented the first DE filaments treated with HTS-type finish.) This improvement is due, at least in part, to the practice of discarding a portion of the roving from the inside and outside of each cake package in which sizing content is most variable. The variability at these positions is due in part to migration effects; these effects occur while the size is liquid, prior to and during the oven bakeout cycle of the cake packages. Volatile losses are probably the highest on the outside of the cake packages, since that surface is in direct contact with heated air during the oven cycle.

As with ignition loss, weight per lineal yard has been very consistent for all lots of roving. The maximum weight range within any given experimental lot has been 0.015 g. This represents a 50% improvement over the previous HTS-E roving, for which a range of over 0.030 g was usually measured.

b. Prepreg

For the impregnation of all experimental lots of material, no deviation was allowed in the basic impregnation process except as required to maintain a uniform resin content. The main impregnation processing parameters and their average levels were:

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Parameter	Average Level
Tower temperature	360 ±10°F
Processing speed	65 ±10 ft/min
Creel tension	750 g
Resin-gel time	4 ±0.25 min

The main parameters which did require adjustment between lots were the specific gravity of the resin bath and creel tension. Resin-bath specific gravity is the most influential factor in the determination of prepreg resin content. A change of 0.005 units of specific gravity causes a 1.0% change in resin content for 20 E HTS roving, with a ribbonization of 2 or 3, assuming otherwise constant resin pickup characteristics for the roving. The influence of creel tension can be explained by reference to Figure 2, which is a schematic diagram of a typical preimpregnation system. As shown in Figure 2, there are only two contact points at A and B prior to the oven. At E and F, work is applied to the roving, and the strand will tend to spread and break down into individual ends. Roving surface area will become larger as the number of unbonded ends becomes larger, and, other things being equal, the resin content will also become greater, since it is dependent upon the surface area of roving exposed to the resin in the impregnation bath. Therefore, resin pickup will vary directly with the amount of debonding that occurs at points A and B. The effect of creel tension is that, as it is raised, a greater amount of work will be applied to the roving at these contact points, and, therefore, a greater amount of debonding can occur. To sum up, resin content will be dependent on roving surface area at time of impregnation; this, in turn, is a function of degree of end-to-end bond, number, and type of contact points and creel tension.

U.S. Polymeric has indicated that variability in end-to-end bond is the most important single roving characteristic responsible for variability in resin content. End-to-end bond may be defined as the amount of work required to break a single 20-end strand of roving into 20 single-end strands. If this property is variable, it is possible that during preimpregnation the degree of end-to-end bond breakdown will also be variable. As noted previously, little work is applied to the roving in the U.S. Polymeric process. The basis

for eliminating work is the assumption that under these conditions the minimum amount of damage will be done to the roving. However, since it is impossible to eliminate all work from the process, debonding does occur for weakly bonded roving. It does not occur as much for strongly bonded roving. At present there is no method for measuring end-to-end bond and therefore no quantitative analysis of the effects of this property is possible. (The Owens-Corning ribbonization measurement does not consider end-to-end bond, since no method is incorporated in the test to apply a controlled and reproducible amount of work to the roving.)

Zero-ribbonization roving has been suggested as one means of eliminating the end-to-end bond problem since this type of roving, having no end-to-end bond, would have a controllable and reproducible surface area. However, it is important to note that any degree of ribbonization is acceptable from the standpoint of resin content uniformity if both the end-to-end bond and the work applied during the impregnation process are both controllable and reproducible. Experience with several preimpregnators who have used zero-ribbonization roving has indicated that the potential improvment in resin content control with this roving may be superseded by other less desirable characteristics. It appears that there is a tendency for catenary and twist buildup to occur during impregnation or zero-ribbonization roving. Normally, these effects are resisted by the end-to-end bond in standard roving. It has been reported that there is a rather high degree of twist and cross-over in zero-ribbonization roving and an apparent lack of it in standard roving. Since the roving forming process is the same for both materials, it would appear that the end-to-end bond in standard roving simply covers up these defects rather than being defect-free, as is normally assumed. However, if a zero ribbonization roving absolutely free of twist and crossover could be supplied, it would result in a prepreg with improved band width and resin-content control.

An alternative to zero-ribbonization roving would be to apply sufficient work to the roving during impregnation to overcome the maximum degree of end-to-end bond that might exist in the input roving. The potential damage to the roving would have to be considered in any attempt to solve the problem in this manner. However, both this approach, as well as zero-ribbonization

roving, are being investigated in the current program.

Thus far, two parameters which affect resin content have been discussed. One factor, specific gravity, is related to the preimpregnation process; the other factor, end-to-end bond, is related basically to the roving but is also affected by the preimpregnation process. Two other roving properties, which affect resin content and which can be treated quantitatively, are weight per yard and sizing content. However, as will be shown, these properties explain only a small part of the resin content variation of prepreg. If a constant resin pickup is assumed, a variation of ±0.03 g (±5%)/yd of roving can account for a resin content variation of about ±0.6%. However, as was noted previously, the total range of weight per yard in most of the experimental lots of roving was only 0.015 g, whereas the range in resin content was 0.9% for the best lot (Lot 7) and between 2 and 3.5% for all the other lots. It should also be noted that extreme care could be exercised in the impregnation of the experimental roving since only one or two rolls of roving were coated at any one time, compared to as many as 50 rolls under standard production conditions.

The contributions of sizing content to resin-content variation is even smaller, since a variation of ±0.5% in sizing content can cause a variation of only ±0.06% in resin content. Actually Lot 7, which had the lowest resin content variation, had one of the highest sizing-content variations of all the experimental lots of roving. Therefore, quantity of sizing and its variability is not a factor of major importance.

Other roving properties which may affect resin content are twist and crossover, affinity of the resin to the finish (wettability), degree of bond between filaments within an end, and degree of penetration of the finish by the resin. There may be other properties which might even be more important than any of those suggested; however, the important factor for all the properties in this group is that no quantitative value can be assigned to any of them at this time, and therefore no quantitative analysis is possible. That current quality control at Owens-Corning does not measure a property directly related to resin pickup characteristic is indicated by the data in Table 5; this table shows the lot averages, the average mean deviation, and the high and low value for the

II Phase I - Study of Roving Forming Process, E (cont.)

standard quality-control tests that are currently applied by Owens-Corning to 20 E HTS roving. This data is for the experimental roving supplied for this program. At least one measurement of each property was made for every roll of roving. In addition to these properties, measurements of catenary, ribbonization, and yardage per pound were also made by Owens-Corning. Catenary and ribbonization data are not included in Table 5 because Owens-Corning data indicated that the former property was invariant at a level of zero and that the latter property was invariant at a level of 2 or 3. (Yardage per pound is essentially the same as weight per yard, which was discussed previously.)

Although several of the properties shown in Table 5 may be eliminated a priori as being related to resin pickup, such properties as wetout rate, stiffness, solids content, and volatile content could conceivably affect prepreg resin content. However, when these properties are compared to the prepreg resin content data also shown in Table 5, no correlation or trend is apparent.

c. Chamber Data

Although in general the hoop filament stress at burst of the 18-in.-chambers has been relatively high, few of the chambers have duplicated the high glass stresses of the simple tensile tests. In the fabrication of an 18-in.-chamber, there are a number of variables which do not have a major effect on simple tensile testing but which can affect chamber performance. Filament alignment, winding pattern, tension uniformity, resin flow, air occlusion, and mandrel contour are just a sampling of the parameters which can adversely affect chamber burst strength. Theoretically the strength in pure tension of the input roving should be the maximum burst stress that can be expected of a chamber, and the difference between this value and the filament stress at burst should indicate the efficiency of the fabrication process. In Figures 3 and 4, chamber filament stress at failure is shown in terms of efficiency factors based on input glass strength. In Figure 3, the efficiency factor was obtained by dividing ultimate hoop filament stresses by lot-average NOL-ring strength. In Figure 4 the efficiency factor used is ultimate hoop filament stress divided by Aerorove strand strength. It is apparent from Figures 3 and 4 that over 80% of

the chambers failed at efficiency factors of 0.85 or higher. Although these figures do not indicate a perfect correlation between ultimate chamber filament stress and either strand or NOL-ring strength, it does point out those chambers for which a serious processing deviation probably occurred. In a positive sense, the figures also indicate that at least 85% of the NOL-ring or strand strength can be expected in a biaxially loaded chamber.

F. CONCLUSIONS

The study to date has produced the following significant conclusions:

1. The Lot 1 improved package has resulted in a higher average dry-glass strength. This conclusion is substantiated by the strength of current production prepreg. As shown in Table 6, the average lot strength of production prepreg for the period between June 1962 and September 1962 was between 367,000 and 400,000 psi. Just prior to this period the average lot strength was about 340,000 psi. These values indicate a significant upward trend, especially when compared to an average prepreg strand strength of between 260,000 and 300,000 psi for production prepreg received during early 1962. The original contention of this program that improvements to the roving forming process and more intensive quality-control procedures would recover a major fraction of the virgin-glass strength appears to be validated by the strength of current production roving.

2. The preimpregnation process does not materially damage roving, but it does result in fabrication material which produces composites equivalent to or better than those produced by the in-process or wet-impregnation process.

3. Current methods for measuring the tensile strength of glass in a composite require modification and optimization before truly quantitative analysis can be made of the efficiency of chamber processing, design, and the influence of resin properties.

4. A method for measuring the resin pickup characteristics of roving is highly desirable in order to guide the development of prepreg with improved resin-content control and wetout characteristics. There are several roving properties of importance in this regard, and probably each should be treated independently. Of major importance would be methods to measure end-to-end bond,

twist-and-crossover, and the variability of finish to wetting.

5. Zero-ribbonization roving would probably permit improved control of prepreg resin content and bond width. However, to be used successfully, twist and crossover would probably have to be eliminated from the roving and careful control would be necessary during impregnation in order not to introduce fiber misalignment. An alternative approach would be to form prepreg directly from single end strand, utilizing dies to form a band of uniform thickness and width.

6. Preliminary work with DE filaments has indicated a potential improvement to be obtained from their use. Although there appears to be no strength advantage in filaments of smaller diameter, resin-content control, and composite uniformity may be improved.

III. PHASE II - PREIMPREGNATION VARIABLE STUDY

A. PROGRAM OUTLINE AND TEST PROCEDURES

The object of the revised Phase II program was to obtain a clearer and more quantitative understanding of the effects of preimpregnation processing parameters on the properties of prepreg roving. The program was divided into four main subtasks, each containing several related parts. These subtasks were process-variable study, resin-content study, band-width study, and processability upon aging. The types of information to be obtained from the work were the following: (1) Definition of the variation of resin content and band width attributable to variation in the preimpregnation parameters, (2) determination of the sensitivity of prepreg filament winding characteristics to variations of preimpregnating processing parameters, (3) definition of both the maximum and minimum degree of polymerization necessary for satisfactory winding, and (4) the effect of degree of polymerization on horizontal shear strength, NOL-ring tensile strength, strand strength, volatile content, etc.

A production lot of 20-end ECG 140/HTS roving was used for Phase II. This material is representative of the improved, high-strength glass roving in which the changes developed in Phase I were incorporated in a production run. The material was inspected for quality control on a sampling basis prior to

III Phase II - Preimpregnation Variable Study, A (cont.)

impregnation.

The resin system used for the Phase II program was the Shell 58/68R in place of E-787 resin employed for the development work in Phase I. This change was due to the proprietary nature of the E-787 composition. Since Phase II was basically a study of preimpregnation variables, it was necessary that the actual composition of the resin be known so that processing effects which are attributable to the resin system would be capable of interpretation. The Shell 58/68R resin system is formulated as follows:

EPON 828	50 parts
EPON 1031-B80*	61.5 parts
MNA (methyl nadic anhydride)	90 PHR
BDMA (benzyldimethyl-amine)	Amount varied for the process variable studies

A single lot of the above components was used to prepare batches of resin mix for the impregnation runs. Except for runs made on the same day with the same resin mix, fresh batches were prepared for all other runs just prior to impregnation.

1. Process Variable Study

A total of 16 runs was made to investigate seven variables which affect the processing characteristics of prepreg. Five of these variables with the region of study are tabulated below:

Process Variable	Region of Study
BDMA content, PHR	0.35 to 0.75
Running speed, ft/min	55 to 83
Tower temperature, °F	340 to 390
Resin solution, sp. gr. at 75°F	0.833 to 0.863
Resin bath temperature, °F	65 to 75

* 80% solution of EPON 1031 resin in MEK.

III Phase II - Preimpregnation Variable Study, A (cont.)

The study included investigation of at least three levels of each of the above process variables, except for the resin-bath temperature, in which two levels were investigated. Each parameter was investigated alone for its effect by maintaining all other parameters constant under a fixed processing condition. To a minor extent, interactions between the different parameters were investigated by changing two or more of the variables within the same run. The two remaining variables, creel tension and package geometry, were varied between rolls of roving within each run but held constant within a given roll.

For each run a set of parameters was predetermined and the conditions were held constant throughout the run. Variations in processing conditions were recorded and allowed to vary within their range of normal variation unless an unusually high variation occurred. When this happened, material prepared up to that point was discarded and the run restarted. Each run consisted of impregnating 10 rolls of roving for a total of 40 lb of prepreg. In addition, three control rolls were processed concurrently to check for resin pick-up, volatile content, and resin flow while in operation. The standard 3-1/8-in. waywind package was utilized for nine rolls of prepreg. The tenth roll was fabricated with a thread-wound pattern and a lead of 0.166 in. (60 turns per 10-in. core length). Five of the waywind rolls and the thread-wound roll were prepared at a tension of 2 lb applied at the creel. Of the other four rolls two waywind rolls each were prepared at a tension of 1.5 and 4 lb.

Changes during a run which occurred in the processing condition were followed by obtaining the gel time, specific gravity, and viscosity of the impregnating resin, and the temperature of the oven prior to, immediately after, and at 1/2-hr intervals during a run. At the start of the program two thermocouples were installed in each of the three towers. With a tower thermostat control and temperature recorder, three temperature readings were taken at predetermined time intervals as noted above. Thermocouples were located at 1/4 the distance from both ends of the tower so as to obtain a temperature profile of the tower.

2. Resin Content Study

Two types of devices were employed in an effort to thoroughly wet the roving and minimize variability of resin content in prepreg. One device, as shown in Figures 5 and 6, consisted of three rollers mounted on two end plates and was designed to apply pressure and to work the roving while immersed in the resin bath. The other device (Figures 7 and 8) was intended to pre-wet the roving and to permit resin penetration as it passed over a wet roller under a light pressure applied at the creel.

3. Band Width Control

This task was included to develop a procedure for measuring the band width of prepreg and to establish the dependence, if any, of band width upon the various parameters used in producing prepreg in the process variable study.

4. Processability on Aging

Rolls of prepreg from selected material lots were investigated for aging characteristics to establish the range of resin polymerization, as determined by viscosity index, necessary for satisfactory winding and its correlation with composite strength properties. A prepreg material was exposed to controlled atmospheric conditions, and at predetermined time intervals gel time and viscosity measurements were taken. Concurrently, composite test specimens which included NOL rings for both tensile and horizontal shear strength, were fabricated and the winding characteristics and physical changes of prepreg were observed.

5. Tests Applied to Prepreg

All incoming lots of prepreg material were inspected for quality control by the standard acceptance procedures. The tests included for all material lots were the determination of gravimetric data (volatile, resin content, and weight per linear yard of roving), strand tensile strength, and resin flow. Gel time and viscosity index tests were applied to determine the effects and sensitivity to variations in preimpregnating processing parameters. These two

tests were also applied to obtain aging data of prepregs exposed to a controlled atmospheric condition and for correlation with respect to prepreg winding characteristics. Procedures for the determination of viscosity index and gel time for prepregs are described in Appendixes A and B, respectively.

Band width measurements and resin content determinations within a roll were made in five locations spaced equally within a roll. A procedure developed for measuring the band width of prepreg roving is described in Appendix C.

B. DISCUSSION OF TEST RESULTS

In order to evaluate prepreg material produced under various manufacturing parameters and to analyze the significance of the results obtained, a test method was necessary by which the processability of the prepreg could be measured quantitatively. The term processability as applied in this report is a generalized term which covers such diverse prepreg qualities as tack, greenness, fray, brittleness, etc., which are properties related to the resin. It is dependent upon three interacting properties, prepreg resin content, solvent content, and degree of polymerization. If these three properties are at a fixed level and nonvariant, the processability characteristics of the prepreg are nonvariant.

Standard methods exist for the measurement of resin and solvent content. However, a test method for degree of polymerization of prepreg resin was only developed recently.

The resin test method is a kinematic viscosity measurement of resin solution prepared by extraction of resin from prepreg with DMF (dimethylformamide). A detailed procedure established for this test is described in Appendix A. Essentially, the viscosity test is based on the premise that in solutions of the same concentration an increased viscosity is a function of increased degree of polymerization. Resins with higher degrees of polymerization offer a greater resistance to flow through a capillary tube in a viscometer and consequently result in longer efflux times. Figure 9 shows the results of a typical laboratory test that was performed in the development of this test method. In this test a

precise quantity of resin was apportioned in several equal parts and the resin advanced to different degrees of polymerization. Each resin sample was then dissolved completely in an accurately measured quantity of dimethylformamide (DMF). As shown in Figure 9, with known equal concentrations and varied degrees of resin advancement between the solutions, the refractive index remained constant, but the kinematic viscosity of each solution was measurably different. To demonstrate the sensitivity of refractive index to concentration, several known solutions of unequal concentrations were prepared and measurements taken. Figure 10 is a representation of the data obtained and indicates that the refractive index is a linear function of concentration. It was possible, therefore, to control the concentration of resin solution in DMF by means of a refractive index measurement. A viscosity measurement on the resin solution would then indicate relative degree of polymerization.

1. Processability and Aging of Prepreg

In addition to the evaluation of prepreg for the study of manufacturing parameters, processability of prepreg upon aging was investigated. Materials used in this study were those produced for the process variable investigation. These materials included a few lots of prepreg which were relatively green and had high tackiness. As prepreg was aged slowly at room temperature, viscosity and gel time measurements were taken and at the same time composite test specimens were fabricated. By means of this slow aging it was possible to follow the whole range of prepreg processability characteristics and relate these to polymerization changes by means of the viscosity index.

The results of the effects of aging on viscosity, gel time, horizontal shear strength, and NOL-ring tensile strength are summarized in Tables 7, 8, 9, and 10 respectively. For a few selected material lots, correlations of the composite strengths with viscosity were made. Figures 11 to 17 show the changes in viscosity, horizontal shear strength, and NOL ring strength with aging time of the material exposed at room temperature. The figures indicate a definite relationship of composite strength properties with the viscosity index of prepreg. For both horizontal shear and NOL-ring tensile specimens, strength increases gradually with increase in viscosity until an optimum range of viscosity is attained.

III Phase II - Preimpregnation Variable Study, B (cont.)

Beyond this optimum range, strength drops off rapidly, generally at a rate faster than the original rate of strength increase. However, the rate of viscosity increase in this range is slow as compared with the earlier stage of advancement. Because of some fluctuations of test results, the optimum point of viscosity is not clearly defined, but from the number of tests conducted, a tentative range of viscosity for optimum strength properties may be determined - e.g., the data indicate that a viscosity for optimum NOL-ring tensile strength and for the horizontal shear strength is obtained at a range of 1.85 to 1.95 viscosity units and 1.90 to 2.00 viscosity units, respectively. At this viscosity range prepreg is still within a region of good processing characteristics but is approaching a region of low tack.

It should be noted that these relationships hold only for the particular process used to prepare the composites. If a different process were used such as increased addition of heat during winding, the curves would be displaced.

Some of the variation that has been obtained in horizontal shear strength of incoming material for production can be explained by the fact that composite strength is dependent upon the viscosity of prepreg system at the time of fabrication. Qualitative observation of incoming material indicates that the spread in the degree of polymerization of standard prepreg is large enough to affect horizontal shear strength.

No good correlation of the gel time data to processability or to degree of resin advancement in prepreg could be made. Generally, the results were erratic and the changes observed with aging time were small. Further discussion of the gel-time data is presented in the following section of this report.

The effect of aging on strand strength and volatile content of prepreg was also investigated. Past experiences have indicated that, besides the basic glass strength, AeroROVE prepreg strand strength was dependent upon the state of resin advancement at the time of specimen preparation. The results obtained from the first few lots of material, shown in Tables 11 and 12, did not indicate any significant changes with time, and testing was discontinued for the

subsequent aging studies.

Viscosity data in relation to aging for a few selected runs is presented in Figure 18. It can be seen that the shape of all curves for materials containing the same amount of BDMA is very similar. This indicates that for a given BDMA content, the rate of resin advancement or polymerization is relatively constant at a constant temperature. Therefore, any storage life for the material which is used up during B-staging in the oven cannot be recovered.

Figure 19 is a composite curve made from a series of actual aging curves of the type shown in Figure 18. Figure 18 indicates the spectrum of processability changes which prepreg undergoes at room temperature, assuming initially that the prepregs were green or insufficiently B-staged. It should be noted that the optimum range of viscosity for filament winding, as shown in Figure 18, to be between 1.82 and 1.96 centistokes, is tentative since differences in resin content and other associated qualities of the prepreg will alter the handling and winding characteristics.

It should be noted also that the optimum range of viscosity index shown in Figure 19 assumes a constant fabrication process. The fabrication process could be modified such that materials of high viscosity index could be usable. As an example, the material could be modified by application of heat or by other means to reduce the viscosity to make the material applicable for filament winding. However, such a modification of the fabrication process to suit material requirements is undesirable under production conditions from the standpoint of reproducibility, reliability, and performance.

Figure 19 also suggests a possible approach to a reproducible and predictable B-staged material for any degree of resin advancement desired to suit any particular process as well as a prepreg with maximum storage life. This approach would be to produce a material which is slightly under-B-staged and to allow it to advance slowly under controlled conditions until the desired processability properties are obtained. The viscosity index could be used to follow these changes and define the point at which the material has optimum winding properties.

III Phase II - Preimpregnation Variable Study, B (cont.)

2. Process Variable Study

A summary of laboratory test data for all incoming material produced under various processing parameters for this study is presented in Table 13. These same lots of material were subjected to outgoing quality-control inspection at the manufacturer's facility. This test data with the measurements of the process conditions taken during the manufacture of these materials is summarized in Table 14. To simplify and facilitate the analysis of the test results, the data shown in Table 13 has been reduced and combined into appropriate groups to illustrate the effect and sensitivity of various process parameters upon qualities of prepreg. Tables 15, 16, and 17 show the effect of resin content, viscosity, and gel time of a prepreg, respectively. Also, corresponding data is graphically illustrated in Figures 20 through 30.

a. Specific Gravity

Figure 20 shows the effect of specific gravity of the impregnating resin bath on resin content of prepreg. In the region of specific gravity, investigated with all other variables held constant at a nominal level, resin content of prepreg is increased by 0.22% for an increase of 0.001 units of specific gravity as measured with a hydrometer at 75°F. This direct relationship is expected, since the concentration of a solution is a function of specific gravity and greater amounts of resin solids are deposited on a roving for a given volume of solution. Also, with increased concentration, the viscosity of a solution correspondingly increases. This increase will have a direct effect on the amount of resin picked up on a roving.

b. Running Speed

As shown in Figure 21, resin content is also related directly to the speed with which a roving is passed through the impregnation system. The increase in resin content is related to the reduction in drainage time of excess solution as compared to slower speeds. The change in resin content was approximately 0.1% increase for an increase of 1 ft per minute of running speed.

III Phase II - Preimpregnation Variable Study, B (cont.)

c. Flow

Within a range of viscosity satisfactory for filament winding, resin flow is related directly to resin content of prepreg. Figure 22 shows a 1.1% change in resin flow for a change of 1% resin content. The slight deviation of flow data from the curve may be traced to a large difference in viscosity index for the material lots. Slightly higher resin flow is obtained from prepregs which are less advanced (B-staged) than with prepregs of equivalent resin content with a higher degree of advancement. However within the usual range of resin content and degree of polymerization in which prepreg is used, resin content is the major controlling influence on flow.

d. Viscosity

As discussed previously, the viscosity test has been used to measure the relative degree of resin polymerization and has been satisfactorily correlated to the processability of prepreg. The viscosity test was also applied to determine the effect of various amounts of BDMA, running speed, and tower temperature on the B-staging of the prepreg resin. In Figure 23 the effect of tower temperature on the viscosity index of the prepreg is shown. These lots of prepreg were all produced under a constant running speed with the resin containing the same amount of BDMA accelerator. It can be seen that the viscosity index is directly related to the amount of heat in-put during the manufacturing process as measured by the tower temperature. Figure 23 shows that for an increase of 10°F tower temperature, viscosity is raised by 0.044 unit. This is equivalent to one day of aging at room temperature (Figure 18). As noted in Table 14, a considerable temperature spread was obtained within a tower when the temperature was measured at three locations and between the towers. Since the deviation of temperature from the fixed condition was significant, actual measured temperatures were used instead of the fixed condition for plotting the data. An average temperature of the actual readings taken from both the top thermocouple and the control gage of the first two towers was used. Although the temperature of the third tower varied between runs from a nominal condition of 150°F, adjustments were not made for these differences in the calculation of average temperature. Small temperature differences at this low level have a

III Phase II - Preimpregnation Variable Study, B (cont.)

relatively minor effect on viscosity as compared with the temperature effect at elevated temperatures.

Figure 24 shows the effect of running speed on viscosity of prepreg produced under a constant tower temperature. Again, the viscosity change is shown to be related directly to the heat in-put during the manufacturing process. With increased running speed, dwell time in the treater towers is reduced and the resultant effect is indicated by a lower viscosity index: the viscosity change was 0.009 centistoke decrease for an increase of 1 ft per minute of running speed. For this data analysis, viscosity was adjusted to correct for the temperature variations obtained between runs. The relationship obtained in Figure 23 was applied to adjust the data to a nominal temperature of 365°F. The relationship of running speed to tower temperature is such that a variation of 1 ft per minute running speed is equivalent to a 2°F change in tower temperature at a nominal temperature of 365°F. It should be noted, however, that all the data may be applicable only to the particular set of equipment and processing conditions utilized for this study. However, this approach to the development of data for process control has a general applicability.

e. Gel Time

As with the analysis of viscosity data, the effect of tower temperature and running speed on gel time is shown in Figures 25 and 26, respectively. A nonlinear relationship appears to exist for the effect of tower temperature on gel time. At a higher processing temperature the resin is advanced to a higher level of B-stage, and the gel time is correspondingly lowered. However, the rate of change in gel time is reduced considerably as the temperature is increased. The data suggests that the polymerization reaction is highly sensitive to small changes in temperature. The reaction progresses rapidly in the early stages to a level in which resin changes become less sensitive to temperature effects. One possible explanation for this phenomenon could be that the resin at a higher level of B-stage becomes more viscous, and the reaction may be subject to diffusion control rather than kinetic or thermodynamic control. Another possible explanation is that some fraction of the original amount of BDMA is volatilized during the heat treatment. Comparison of the gel-time data for

III Phase II - Preimpregnation Variable Study, B (cont.)

virgin resin mixes (Table 14) and prepregs (Table 16) provides substantial evidence of this behavior. In some cases, longer gel time is obtained from prepreg than for the same resin mix from which the prepreg was made. Also, data to support the contention of BDMA volitilization is shown in Figures 27 and 28. Figure 27 shows that gel time is definitely affected by the amount of BDMA contained in the original resin mix and, even upon aging, the gel time is distinctly different (dependent on BDMA content). Figure 28, correlating gel time with aging, indicates a similar type of dependence of gel time as obtained in Figure 27, but the magnitude of difference was smaller. These prepregs were produced with a resin mix containing the same amount of BDMA but processed under various processing conditions.

The effect of running speed on gel time is shown in Figure 26. As with the viscosity data, adjustments were made to gel-time data to correct for the variation in tower temperature between runs. Based on Figure 25, a correction factor of 0.8 sec per 1°F difference in tower temperature was applied. Even with this adjustment, a large scatter in gel time was obtained. These scatters were especially evident in the aging study.

f. Effect of BDMA

Three levels of BDMA accelerator content in the basic resin formulation were initially investigated to establish the optimum content for prepreg application. Strength properties of the composites and reactivity of prepreg for both aging and storageability were considered for the selection of BDMA content to be used for the remainder of the runs. Data on gel time for various amounts of BDMA is presented in Table 11, and corresponding data is shown graphically in Figure 29. It can be seen that the reactivity of the Shell 58/68R resin system is more sensitive to small changes in the amounts of BDMA at a lower range of BDMA content. Composite strength in horizontal shear is slightly superior with higher BDMA content, but not significantly different in the NOL-ring tensile strength.

g. Correlation of Gel Time and Viscosity

The data indicates that the gel-time test is not so

reproducible or reliable as the viscosity test, and no direct correlation can be made of the gel-time data to the processability of prepreg. However, the value of a gel-time test lies in the determination of the reactivity of a prepreg system and, as such, compliments the viscosity data. Figure 30, showing the relationship of gel time and viscosity, was derived from data presented previously in Figures 23 through 26. The large scatter of points shown in Figure 30 is due in part to the large variance in gel-time data and in part to the deviations of viscosity data. In spite of the scatter, however, the type of curve shown in Figure 30, is useful for material inspection and can be applied for process optimization of prepregs. An example of the type of material inspection which can be applied is shown in Figure 30 for a prepreg containing 0.75 PHR BDMA. In this case it is noted that the gel time is considerably lower than is usual for the corresponding viscosity. This would immediately show that the material is too reactive and that storageability or bench life of the material would be appreciably shortened.

3. Resin Content Study

In order to reduce the variability of resin content in prepregs, special devices were developed for this program and applied as described previously in the section on test procedure. Laboratory test data of the two runs produced by utilization of these devices is summarized in Table 18. Based on standard deviation of 0.48 and 0.34% as compared with 0.43% of the standard-type material lots (Table 15), the variability of resin content between rolls is not improved significantly from other material lots produced without the special devices.

Table 19 summarizes the data of resin content in prepreg for various tensions applied at the creel. The data shows a slight trend to a higher resin content with 1.5-lb creel tension as compared with 2.0 and 4.0-lb creel tension, especially for process conditions used to produce high resin content (above 23%) prepregs. Generally, however, the results were inconsistent. In order to gain a better understanding of the effect of tension on resin content and the inconsistencies obtained, roving tensions were measured at various locations between contact points while in operation. These measurements, summarized

in Table 20, indicate that tension introduced at the creel is immediately altered after the first contact point (as shown in Figure 2 for the schematic diagram of the preimpregnation line) and that tension varies with time within the same location. If roving tension is one of the contributing factors which affect the resin content in prepregs, variations in roving tension as shown in Table 20 may be a partial explanation for the variations observed within a material lot and even within a roll.

4. Package Geometry

For each impregnation run one of the 10 prepreg rolls was wound with a thread-wound pattern with a lead of 0.116 in. as compared with a 3.125-in. lead of the standard waywind package. The objective of this study was to determine whether any improvement or benefit is gained from a reduced packaging lead angle over the standard pattern. Comparative results of the AeroROVE strand test and fiber tensile strength calculated from the NOL-ring composite tensile strength are presented in Table 19. Strengthwise, there is no significant difference between the two packages, but uniformity in band width within a roll is superior with a thread-wound package. No sudden band width changes, such as those which occur at the traverse termination points of a waywind package, were observed, and no unusual winding problem was experienced during the specimen fabrication. However, the package had a tendency to flow out and deform the package shape under a slight take-up winding tension, more so than for a standard waywind package. This condition was more clearly evident when winding a "green" prepreg. It was not possible to evaluate the influence of this factor (package deformation) on processability and prepreg quality during this program.

5. Band Width

A relatively simple procedure has been developed, as described in Appendix C, for the measurement of the band width of prepreg roving. Three measurements of band width were taken for each specimen as follows: (1) width as unspooled directly from a package to represent the dependence upon the roving and the processing parameters used to produce prepreg, (2) width under tension to represent a fabrication condition where winding tension is normally applied,

and (3) width at the table edge under tension to represent a condition immediately after being applied on the part. Band width measurements between rolls and within single rolls of prepreg are shown in Table 21. A definite change in band width is noted for the three conditions of measurement. The amount of change appears to be related directly to resin content and inversely to the viscosity index of the prepreg. Since prepreg processing variables affect both resin content and the degree of polymerization, it may be concluded that band width roving is dependent upon the various parameters used in producing prepreg.

C. CONCLUSIONS

The processing characteristics of prepreg have been shown to be very sensitive to levels of manufacturing parameters used in producing prepregs. Test data shows that resin content is affected directly by process variables such as specific gravity of the impregnating resin solution, running speed, and, to some extent, tension applied at the creel. Also, bandwidth is affected by variations in both resin content and degree of polymerization, which in turn is dependent on the processing parameters. The most significant single property of prepreg which affects processability is the degree of resin polymerization (B-staging) if the resin and solvent content are relatively constant. Manufacturing parameters which affect this property are tower temperature and running speed, both of which are related to the amount of heat-energy input to the material during the manufacture. However, in spite of these relationships and their sensitive dependence on levels of process variables, very uniform and predictable prepregs can be made if manufacturing parameters are closely controlled and maintained.

An approach to setting quantitative limits for prepreg manufacturing has been demonstrated by the application of the viscosity-index test as a means of assigning differences in processing characteristics of prepregs. This study has also demonstrated the critical need for measuring degree of polymerization during the early stages in the application of any new resin to prepreg. Although levels of manufacturing parameters defined in this program are for a specific process, this approach has general applicability to other prepreg manufacturing processes.

III Phase II - Preimpregnation Variable Study, C (cont.)

This is the first time that quantitative data of this type defining the manufacture of materials, has been developed, and it is considered a significant breakthrough in the development of scientifically controlled processes and materials in the field of reinforced plastics. Further studies recommended in this area of development would be investigations of interactions between prepreg processing variables and relationships of prepreg processability to the strength of filament-wound internal pressure vessels.

TABLE 1

Table 1 a

GRAVIMETRIC AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

Lot No. 1

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av.	High	Low	Loss on Ignition (%) Av.	High	Low	Weight Per Yard (Glass) (g) Av.	High	Low	NOL Ring (Glass Stress), (ksi) Av.	High	Low
1	356.6	373.3	319.5	1.45	1.51	1.36	0.656	0.659	0.653	330.0	346.6	311.9
2	345.7	363.6	318.2	1.47	1.50	1.34	0.660	0.661	0.659	321.0	345.0	308.8
3	357.4	382.4	343.3	1.38	1.50	1.35	0.661	0.662	0.659	330.3	339.5	313.4
4	362.5	390.9	312.5	1.35	1.35	1.35	0.661	0.662	0.658	329.4	352.0	310.0
5	343.4	371.6	329.7	1.46	1.49	1.33	0.666	0.669	0.662	321.0	344.3	308.8
6	301.6	315.0	293.3	1.25	1.35	1.20	0.662	0.664	0.659	350.6	358.3	340.2
7	345.5	373.9	281.3	1.33	1.36	1.21	0.656	0.657	0.656	318.1	336.0	301.8
8	363.7	375.9	317.9	1.35	1.50	1.20	0.660	0.661	0.659	[illegible]	337.1	315.9
9	342.5	370.5	296.6	1.32	1.35	1.20	0.660	0.662	0.658	324.2	337.9	316.8
10	352.5	380.1	329.9	1.46	1.49	1.39	0.664	0.664	0.662	330.2	349.1	314.8
Lot Average	347.5			1.38			0.660			327.3		
Av Deviation	11.3			.06			0.003			6.2		

Table 1 b

Lot No. 2

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av.	High	Low	Loss on Ignition (%) Av.	High	Low	Weight Per Yard (Glass) (g) Av.	High	Low	NOL Ring (Glass Stress), (ksi) Av.	High	Low
1	283.7	[illegible]	268.0	1.62	[illegible]	1.06	0.655	0.657	0.653	320.7	[illegible]	[illegible]
3	278.8	304.1	247.9	1.70	1.96	[illegible]	0.651	0.654	0.648	311.6	[illegible]	[illegible]
4	299.0	311.1	[illegible]	1.97	2.56	[illegible]	0.651	0.653	0.647	322.1	[illegible]	[illegible]
5	312.8	351.6	[illegible]	0.78	1.05	0.70	0.657	0.659	0.656	259.8	313.0	[illegible]
6	308.2	330.8	287.9	2.10	2.54	1.80	0.653	0.654	0.651	302.1	[illegible]	[illegible]
7	325.3	362.9	304.1	2.48	2.69	[illegible]	0.651	0.652	[illegible]	[illegible]	[illegible]	[illegible]
8	301.1	332.8	257.6	0.33	[illegible]	0.15	0.658	0.659	0.657	[illegible]	[illegible]	[illegible]
9	379.4	408.5	365.8	1.81	2.11	1.51	0.650	0.651	0.649	[illegible]	[illegible]	[illegible]
11	330.1	350.8	[illegible]	1.00	[illegible]	0.76	0.652	0.653	0.651	[illegible]	[illegible]	[illegible]
12	330.6	365.0	294.9	1.46	1.67	1.27	0.646	0.647	0.646	[illegible]	[illegible]	[illegible]
Lot Average	315.8			1.48			0.652			[illegible]		
Av Deviation	22.0			.47			0.003			[illegible]		

Table 1 c

Lot No. 3

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av.	High	Low	Loss on Ignition (%) Av.	High	Low	Weight Per Yard (Glass) (g) Av.	High	Low	NOL Ring (Glass Stress), (ksi) Av.	High	Low
1	344.4	394.0	292.9	1.36	[illegible]	1.36	0.653	0.654	0.651	[illegible]	[illegible]	320.7
3	336.8	379.6	276.1	1.54	1.65	1.50	0.652	0.656	0.649	[illegible]	[illegible]	[illegible]
4	373.3	399.5	300.9	1.47	1.51	1.36	0.653	0.655	0.652	[illegible]	[illegible]	[illegible]
5	350.0	365.2	336.8	1.52	1.52	1.51	0.651	0.653	0.650	[illegible]	[illegible]	[illegible]
6	364.0	405.0	311.6	1.37	1.77	1.36	0.650	0.653	0.648	[illegible]	[illegible]	[illegible]
7	354.8	385.4	313.4	1.77	1.38	1.77	0.646	0.648	0.645	[illegible]	[illegible]	[illegible]
8	346.3	387.2	289.6	1.39	1.37	1.77	0.647	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9	367.4	394.4	[illegible]	1.51	1.54	1.39	0.639	0.640	[illegible]	[illegible]	[illegible]	[illegible]
11	357.2	379.2	336.3	1.77	1.38	1.36	0.648	0.651	0.645	[illegible]	[illegible]	[illegible]
12	357.2	353.6	273.9	1.55	1.66	1.52	0.647	0.651	0.645	[illegible]	[illegible]	[illegible]
Lot Average	346.3			1.44			0.648			[illegible]		
Av Deviation	13.7			0.07			0.003			[illegible]		

TABLE 1 (cont.)

Table 1 d

GRAVIMETRIC AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

Lot No. 4

Roll No.	Ultimate Tensile Strength (Stress) (ksi) Av	High	Low	Loss on Ignition (%) Av	High	Low	Weight Per Yard (Glass) (g) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
2	339.5	364.0	301.2	1.61	1.68	1.52	0.645	0.645	0.644	330.6	337.1	[illegible]
4	344.4	396.7	320.1	1.50	1.59	1.48	0.656	0.658	0.655	[illegible]	[illegible]	[illegible]
5	337.6	375.0	290.2	1.49	1.52	1.37	0.646	0.647	0.646	[illegible]	[illegible]	[illegible]
6	335.1	380.8	331.0	1.48	1.52	1.46	0.648	0.650	0.647	341.7	343.7	[illegible]
7	323.1	339.0	297.9	1.40	1.50	1.34	0.657	0.658	0.656	334.9	345.8	[illegible]
8	329.9	344.6	317.0	1.41	1.50	1.35	0.653	0.656	0.652	[illegible]	[illegible]	[illegible]
10	328.4	350.9	303.9	1.35	1.36	1.35	0.654	0.655	0.652	[illegible]	342.9	322.0
11	346.4	363.6	328.2	1.31	1.35	1.20	0.656	0.658	0.655	[illegible]	357.6	342.1
12	342.7	363.1	314.6	1.37	1.37	1.36	0.646	0.648	[illegible]	[illegible]	343.3	334.6
Lot Average	339.4			1.44			0.650			[illegible]		
Av Deviation	7.7			0.07			0.004			10.1		

Table 1 e

Lot No. 5

Roll No.												
2	320.0	347.8	294.5	1.64	1.67	1.52	0.647	[illegible]	[illegible]	[illegible]	342.5	[illegible]
3	315.7	397.1	283.4	1.54	1.66	1.51	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
4	315.8	345.9	287.9	1.56	1.68	1.52	[illegible]	0.647	[illegible]	[illegible]	[illegible]	[illegible]
5	333.8	386.0	296.5	1.50	1.53	1.39	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
6	372.4	393.2	352.8	1.50	1.54	1.39	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
7	335.9	380.4	313.5	1.53	1.67	1.38	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
8	368.1	400.9	335.2	1.52	1.52	1.52	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
9	360.0	395.5	318.8	1.44	1.51	1.35	0.655	0.647	0.653	334.9	344.8	[illegible]
12	350.2	378.7	327.5	1.54	1.65	1.36	0.654	0.655	0.653	[illegible]	[illegible]	[illegible]
13	385.7	392.7	332.6	1.54	1.65	1.51	0.654	0.654	0.652	322.3	334.5	[illegible]
Lot Average	346.7			1.53			[illegible]			332.6		
Av Deviation	17.7			0.09			0.004			[illegible]		

Table 1 f

Lot No. 6

Roll No.												
1	284.9	349.2	252.7	1.36	1.36	1.36	0.653	0.653	0.652	315.3	321.2	[illegible]
3	293.5	308.1	279.6	1.25	1.37	1.21	0.649	0.651	[illegible]	300.1	[illegible]	279.3
9	285.3	300.9	261.9	1.21	1.21	1.21	[illegible]	0.654	0.652	303.8	[illegible]	[illegible]
11	352.9	387.2	336.2	1.46	1.52	1.38	0.647	0.647	[illegible]	[illegible]	[illegible]	[illegible]
14	292.8	314.8	273.1	1.37	1.38	1.36	0.648	[illegible]	[illegible]	297.1	[illegible]	[illegible]
17	327.0	351.3	291.2	1.34	1.37	1.22	0.649	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
21	317.2	350.2	293.0	1.30	1.37	1.21	[illegible]	0.653	[illegible]	[illegible]	322.4	311.9
22	333.5	355.4	295.4	1.40	1.52	1.36	0.650	[illegible]	[illegible]	311.6	332.5	[illegible]
24	300.0	309.3	286.2	1.27	1.37	1.21	[illegible]	0.651	0.647	[illegible]	[illegible]	[illegible]
25	325.4	341.0	301.1	1.33	1.37	1.21	0.651	[illegible]	[illegible]	[illegible]	323.7	[illegible]
Lot Average	302.4			1.33			0.650			[illegible]		
Av. Deviation	19.9			0.06			0.002			9.4		

TABLE 1 (cont.)

Table 1 g

GRAVIMETRIC AND STRENGTH DATA OF DRY EXPERIMENTAL ROVING

Lot No. 7

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av	High	Low	Loss on Ignition (%) Av	High	Low	Weight Per Yard (Glass) (g) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
3	344.2	373.3	322.2	1.66	1.78	1.63	0.662	0.663	0.659	344.1	372.6	313.8
4	327.9	344.4	299.1	1.53	1.79	1.33	0.661	0.666	0.658	332.2	345.3	325.1
5	327.2	349.3	300.2	1.43	1.66	1.34	0.657	0.659	0.655	316.4	328.2	308.4
6	316.2	345.8	281.7	1.37	1.50	1.35	0.655	0.657	0.654	296.0	300.4	289.7
7	333.5	360.7	291.1	1.36	1.50	0.94	0.656	0.657	0.654	321.5	322.8	319.8
8	338.8	373.3	307.1	1.37	1.47	1.34	0.655	0.658	0.653	317.7	327.1	305.5
9	341.9	361.8	309.8	1.29	1.36	1.20	0.651	0.655	0.649	308.8	326.1	296.5
10	314.3	352.6	282.2	1.35	1.38	1.35	0.653	0.656	0.650	336.1	346.3	320.4
11	361.4	380.7	320.9	1.56	1.80	1.50	0.652	0.654	0.650	336.0	340.1	329.2
12	356.3	375.5	332.0	1.61	1.66	1.57	0.655	0.660	0.651	321.6	328.7	317.4
Lot Average	335.9			1.45			0.655			323.0		
Av Deviation	12.5			0.1			0.003			11.2		

Lot No. 8

Table 1 h

Roll No.												
1	313.1	338.8	275.7	1.60	1.69	1.53	0.642	0.644	0.641	310.3	321.8	304.4
2	294.5	331.6	276.9	1.55	1.67	1.52	0.647	0.648	0.646	310.9	315.2	304.9
5	301.1	328.1	243.7	1.60	1.70	1.53	0.640	0.643	0.637	302.2	308.7	294.9
6	311.6	330.0	294.3	1.58	1.67	1.52	0.650	0.652	0.648	300.9	308.9	295.5
7	290.7	312.8	260.5	1.62	1.68	1.52	0.645	0.647	0.643	297.8	306.0	285.4
8	308.6	338.1	238.3	1.49	1.50	1.48	0.661	0.664	0.658	310.3	314.5	308.1
10	314.6	330.5	265.6	1.62	1.66	1.56	0.658	0.694	0.626	299.2	316.2	271.5
11	311.6	350.8	267.7	1.49	1.52	1.37	0.650	0.650	0.648	311.9	315.9	305.5
12	313.4	337.8	279.8	1.49	1.53	1.37	0.646	0.650	0.644	309.7	329.7	292.6
13	285.2	315.3	243.5	1.40	1.53	1.37	0.647	0.650	0.642	310.5	321.2	296.7
Lot Average	304.4			1.54			0.648			306.4		
Av Deviation	9.2			0.06			0.005			5.1		

TABLE 2

Table 2 a

GRAVIMETRIC AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING

Lot No. 1

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av	High	Low	Loss on Ignition (Resin Solids) (%) Av	High	Low	Volatile Content, (%) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
1	339.4	354.2	310.7	19.48	19.90	19.08	0.92	0.97	0.84	328.5	344.9	313.1
2	322.4	330.7	313.3	20.53	21.08	19.83	0.96	1.08	0.85	345.7	353.1	338.2
3	295.0	315.4	272.3	20.90	21.40	20.55	0.98	1.05	0.95	339.7	344.3	334.0
4	311.1	321.1	289.5	21.68	22.71	20.45	1.13	1.19	1.04	337.0	371.3	297.7
5	351.6	[illegible]	333.3	21.13	22.26	20.24	1.07	1.16	0.95	332.4	347.6	313.5
6	305.0	330.1	289.1	19.41	20.31	18.93	1.20	1.21	1.19	326.7	344.1	315.4
7	310.1	321.7	297.1	19.74	19.88	19.52	1.25	1.33	1.20	335.4	360.2	309.0
8	[illegible]	320.5	356.8	19.42	19.69	19.54	1.24	1.32	1.08	339.5	349.8	332.0
9	318.0	332.6	309.5	19.47	20.19	19.06	0.96	0.97	0.95	323.5	335.7	311.9
10	321.8	346.4	298.2	21.82	23.19	20.44	0.94	0.96	0.92	333.0	338.4	327.9
11	309.4	322.3	290.7	21.61	22.07	21.25	0.98	1.05	0.94	338.1	353.1	331.0
Lot Average	315.7			20.51			1.05			334.5		
Av Deviation	13.4			0.83			0.11			5.2		

Table 2 b

Lot No. 2

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av	High	Low	Loss on Ignition (Resin Solids) (%) Av	High	Low	Volatile Content, (%) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
1	313.4	335.1	275.0	20.80	21.38	19.68	1.51	1.54	1.46	330.2	342.0	319.5
2	[illegible]	345.3	297.3	20.63	20.95	20.12	1.42	1.43	1.42	339.6	346.9	332.2
3	[illegible]	327.4	291.1	19.98	20.40	19.54	1.46	1.54	1.44	346.5	357.1	336.5
4	[illegible]	[illegible]	287.0	19.40	19.54	19.22	1.51	1.58	1.47	325.9	335.7	307.4
5	[illegible]	[illegible]	277.0	18.99	19.66	18.33	1.34	1.55	1.33	352.1	361.5	347.2
6	[illegible]	[illegible]	284.3	17.91	18.35	17.66	1.32	1.48	1.24	346.2	361.3	332.4
7	[illegible]	[illegible]	285.1	19.20	19.74	18.73	1.65	1.71	1.96	333.3	365.6	325.4
8	[illegible]	[illegible]	[illegible]	17.20	17.54	16.88	1.75	1.88	1.62	330.7	340.6	316.5
9	[illegible]	318.6	270.3	18.21	18.38	17.95	1.91	1.48	1.36	345.4	353.6	336.3
10	[illegible]	313.4	[illegible]	17.46	17.93	17.51	1.35	1.48	1.23	312.5	342.8	266.8
11	[illegible]	[illegible]	[illegible]	18.97	19.98	18.33	1.90	1.99	1.44	335.0	352.1	321.7
12	[illegible]	[illegible]	[illegible]	19.78	20.36	18.83	1.66	1.83	1.45	341.0	364.8	335.5
13	[illegible]	313.7	[illegible]	17.98	18.87	16.94	1.86	2.22	1.63	356.9	360.6	353.1
14	[illegible]	[illegible]	271.5	19.12	20.80	18.18	1.74	1.80	1.70	323.8	336.9	309.3
Lot Average	302.3			18.98			1.54			337.0		
Av Deviation	9.3			0.85			0.13			9.7		

TABLE 2 (cont.)

Table 2 c

GRAVIMETRIC AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING

Lot No. 3

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av	High	Low	Loss on Ignition (Resin Solids) (%) Av	High	Low	Volatile Content, (%) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
1	320.3	328.3	295.1	19.25	19.50	18.81	1.85	1.97	1.73	298.8	316.9	275.7
2	315.3	329.9	307.9	18.50	19.00	17.90	1.79	1.83	1.72	321.2	327.1	314.1
4	318.5	344.4	289.1	19.32	19.64	19.04	1.90	2.15	1.78	321.5	337.4	309.6
5	283.0	309.7	248.0	19.26	19.51	18.92	1.78	1.83	1.71	324.6	328.5	317.8
6	314.9	322.6	307.9	19.02	19.48	18.63	1.65	1.72	1.61	311.7	317.8	308.0
7	312.2	318.4	294.5	19.14	19.54	18.85	1.78	1.83	1.70	319.6	322.4	317.9
8	309.2	322.5	299.1	22.17	23.01	21.39	2.26	2.42	2.11	333.3	351.6	315.9
9	308.9	328.4	285.5	19.10	19.78	18.23	1.97	1.99	1.95	338.5	345.5	325.0
10	313.7	339.8	292.8	21.19	22.53	20.47	2.18	2.50	1.65	314.0	320.9	307.6
11	294.3	307.8	269.9	19.60	20.11	18.83	2.09	2.17	2.04	288.9	317.4	274.6
12	338.1	357.6	303.5	20.66	21.09	19.90	1.84	1.91	1.79	300.3	304.5	296.6
13	307.0	330.5	286.8	19.11	20.64	17.55	2.38	2.60	2.14	314.6	340.9	297.5
14	276.2	312.8	251.2	21.10	21.71	20.09	1.80	1.94	1.58	290.1	318.1	274.3
Lot Average	307.9			19.80			1.94			313.6		
Av Deviation	12.3			0.91			0.17			12.0		

Lot No. 4

Table 2 d

Roll No.												
7	324.8	338.9	310.6	18.32	18.48	18.03	1.84	1.96	1.72	328.2	333.2	321.2
8	316.4	324.2	297.5	18.34	18.75	17.86	1.65	1.82	1.48	336.9	366.1	314.3
9	333.3	360.5	316.6	15.08	15.12	14.93	1.71	1.80	1.67	334.8	337.4	330.5
10	317.6	347.8	279.4	19.17	19.48	18.97	1.79	1.95	1.60	338.9	353.2	323.6
11	321.4	341.0	305.3	19.92	20.14	19.87	1.81	1.92	1.69	334.4	343.1	321.6
Lot Average	322.7			18.15			1.76			334.6		
Av Deviation	5.1			1.26			0.06			2.6		

TABLE 2 (cont.)

Table 2 e

GRAVIMETRIC AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING

Lot No. 5

Roll No.	Ultimate Tensile Strength (Strand) (ksi) Av	High	Low	Loss on Ignition (Resin Solids) (%) Av	High	Low	Volatile Content, (%) Av	High	Low	NOL Ring (Glass Stress), (ksi) Av	High	Low
3	302.1	313.4	293.8	19.73	19.92	19.42	1.97	2.18	1.81	342.4	350.5	332.7
4	299.3	328.9	283.9	20.40	21.06	19.90	1.92	1.94	1.90	340.2	355.0	328.5
5	300.7	315.0	285.4	19.00	19.32	18.66	1.83	1.86	1.82	340.8	344.5	334.6
7	342.5	365.8	295.4	18.58	18.97	17.98	1.88	2.07	1.71	342.7	351.9	333.4
8	316.0	337.0	295.0	19.30	19.56	19.05	1.66	1.82	1.46	361.0	368.9	352.8
9	313.5	325.8	301.4	19.09	19.41	18.63	1.88	1.96	1.83	346.8	355.9	334.1
10	316.9	332.4	282.4	19.18	19.68	18.75	1.99	2.08	1.95	350.9	356.1	347.4
11	340.7	361.1	312.5	19.42	19.80	19.08	1.91	2.19	1.70	352.5	358.1	345.2
12	345.4	353.0	340.3	19.88	20.48	19.51	1.62	1.94	1.10	355.4	365.8	339.7
13	334.1	348.3	301.8	19.64	19.87	18.97	1.99	2.07	1.94	355.6	370.0	348.2
14	329.1	356.1	290.8	18.69	18.87	18.56	2.18	2.29	2.06	358.1	363.8	352.7
Lot Average	321.8			19.35			1.89			349.7		
Av Deviation	15.0			0.41			0.11			6.4		

Table 2 f

Lot No. 6

Roll No.												
9	304.8	315.2	294.5	24.19	24.30	24.10	2.53	2.58	2.47	313.6	330.0	305.0
10	297.7	312.9	286.4	25.16	25.44	24.92	2.27	2.35	2.11	329.5	342.0	316.5
11	305.1	318.5	295.4	24.31	24.58	24.18	2.46	2.59	2.31	324.3	337.5	314.4
12	321.9	352.4	288.9	21.10	21.37	20.84	2.02	2.07	1.96	316.3	331.5	297.9
13	310.7	317.6	292.1	22.99	23.10	22.88	2.16	2.25	2.03	310.0	315.9	299.3
14	314.2	326.9	296.8	21.81	21.93	21.68	2.19	2.28	2.11	312.4	317.4	309.8
15	319.1	327.2	306.5	23.92	24.30	23.58	2.34	2.43	2.28	313.3	324.0	298.4
16	306.5	343.3	283.4	22.88	23.24	22.57	2.45	2.48	2.42	304.9	307.9	300.9
17	330.5	345.5	314.5	24.16	24.30	23.89	2.27	2.44	2.15	310.1	324.0	296.4
18	322.2	330.3	314.2	23.48	23.67	23.37	2.09	2.16	1.99	306.1	311.0	299.3
19	317.1	334.4	284.7	22.71	22.86	22.47	1.92	2.04	1.80	308.0	313.9	304.8
20	311.6	363.5	265.4	21.34	21.61	20.96	1.86	1.95	1.77	309.2	318.9	300.6
23	339.4	348.6	318.8	21.31	21.73	21.03	1.80	2.00	1.59	306.7	320.5	300.0
Lot Average	315.4			23.03			2.18			313.0		
Av Deviation	8.8			1.08			0.19			5.6		

TABLE 2 (cont.)

Table 2 g

GRAVIMETRIC AND STRENGTH DATA OF EXPERIMENTAL PREPREG ROVING

Lot No. 7

Roll No.	Ultimate Tensile Strength (Filament) (ksi) Av	High	Low	Loss on Ignition (Resin Solids) (%) Av	High	Low	Volatile Content, (%) Av	High	Low	NOI Ring (Glass Stress), (ksi) Av	High	Low
1	360.8	361.2	359.0	20.48	21.25	19.70	1.36	1.45	1.31	350.1	358.7	338.1
2	342.0	360.2	327.3	20.58	21.12	20.23	1.34	1.43	1.28	349.4	358.8	344.4
3	341.4	359.5	325.2	19.88	19.95	19.85	1.63	1.67	1.56	345.0	356.8	335.8
4	337.9	353.8	311.3	19.96	20.50	19.49	1.45	1.81	1.20	347.8	355.7	338.9
5	325.4	350.9	293.4	20.00	20.54	19.30	1.95	2.62	1.43	343.7	355.3	332.7
6	344.6	364.1	333.1	19.67	20.07	19.38	2.42	2.49	2.38	321.8	325.0	319.6
8	332.1	339.6	319.1	20.17	21.26	19.99	1.95	1.90	1.43	346.5	352.0	343.2
Lot Average	340.3			20.10			1.68			343.5		
Av Deviation	7.9			0.26			0.29			5.8		

Table 2 h

Lot No. 8

Roll No.												
1	340.8	356.4	320.3	19.12	19.49	18.87	1.26	1.30	1.23	358.5	363.4	354.8
2	288.1	293.6	281.5	23.14	23.51	22.89	2.32	2.40	2.22	356.5	364.8	348.4
3	289.4	324.2	260.3	22.81	23.08	22.55	2.68	2.71	2.66	350.2	357.7	342.8
4	299.8	308.1	281.9	21.45	21.74	21.22	2.23	2.30	2.14	354.7	355.3	353.8
5	272.8	280.2	261.2	23.25	23.42	23.10	2.49	2.54	2.42	349.3	351.2	348.2
6	279.0	308.8	251.9	23.11	23.48	22.78	2.54	2.62	2.47	341.2	347.6	325.5
Lot Average	298.0			22.14			2.25			351.7		
Av Deviation	17.5			1.24			0.17			5.8		

TABLE 3

18-in. DIAMETER FILAMENT WOUND CHAMBERS - TEST RESULTS

Chamber Number	Material**	Burst Pressure (psi)	Hoop Filament Stress-Cylinder (ksi)	Long. Filament Stress-Cylinder (ksi)	Composite Stress-Cylinder (ksi)	Long. Composite Stress-Cylinder (ksi)	Long. Composite Stress-Head (ksi)	Strand* Strength (ksi)	NOL Rings* Strength (ksi)
0090-14S	Lot 37-W***	846***	305.9	245.5	127.8	63.6	165.7	297.0	-
0090-16A	Lot 37-W	895	311.2	235.2	126.6	63.3	158.8	337.0	-
0525-3	Lot 1-W	860	267.1	206.2	113.4	56.7	152.7	363.7	320.5
0525-4	Lot 37-P	760	260.0	200.2	104.2	52.1	132.2	333.3	323.0
0525-5	Lot 1-P	950	297.8	236.3	120.8	60.4	156.2	311.1	337.1
0525-6	Lot 3-W	910	306.9	231.6	124.4	62.2	156.1	333.3	297.8
0525-7	Lot 2-W	980	322.1	249.7	132.2	66.1	168.5	285.3	323.4
0525-8	Lot 4-W	890	294.1	235.8	121.0	60.5	159.2	344.4	324.0
0525-9	Lot 5-W	870	307.8	212.8	120.6	60.3	143.6	355.7	321.0
0525-10	Lot 3-P	930	339.3	232.4	126.8	63.4	153.6	338.0	318.5
0525-11	Lot 2-P	900	297.7	238.8	118.8	59.4	153.9	292.2	344.9
0525-12	Lot 4-P	852	280.1	236.0	112.9	56.5	148.7	316.4	333.8
0525-13	Lot 4-P	790	267.4	208.0	106.4	53.2	137.5	324.9	327.7
0525-14	Lot 5-P	980	337.8	252.3	132.1	66.6	166.1	342.5	342.7
0525-15	Lot 7-W	875	287.1	216.7	115.3	57.7	146.3	327.9	331.7
0525-17	Lot 7-P	900	303.1	234.5	118.6	59.3	152.7	341.4	345.1

* Values are for roll of roving from which chamber was made.

** W = wet winding; P = prepreg.

*** All chambers were weak hoop and failed in the hoop wraps. All chambers were CSO design. Test pressure prior to burst was 400 psig for one minute.

TABLE 4

EFFECT OF VINYL COATING ON PREPREG STRAND STRENGTH

Lot No.	Roll No.	Ultimate Tensile Strength (psi x 10^{-3})					
		Standard Prepreg Strand			Vinyl-Coated Prepreg Strand		
		Average	High	Low	Average	High	Low
3-804	19	293.4	310.2	272.4	377.2	391.2	352.0
4-826	7	324.8	358.9	310.6	393.1	406.3	371.5
5-838	12	345.4	353.0	340.3	381.2	408.6	338.0
7-846	7	353.1	365.8	335.8	399.7	412.0	377.3

TABLE 5

OWENS-CORNING QUALITY CONTROL DATA FOR EXPERIMENTAL GLASS

Lot No.	Stiffness	Volatile Content (%)	Solids Content (%)	Wet-Out Rate	Package Hardness	Fuzz Content (x10)	Resin Content (%)		
							Avg.	High	Low
1	3.88±0.11	0.204±0.010	1.12±0.03	89.50±1.50	66.1±3.5	0.08±0.045	20.51±0.84	21.82	19.47
2	3.20±0.11	0.015±0.004	1.21±0.07	89.70±1.48	68.6±1.7	1.54±0.65	18.98±0.85	20.80	17.20
3	3.57±0.07	0.106±0.015	1.19±0.03	86.76±2.16	69.2±1.7	1.64±0.60	19.83±0.95	22.17	18.50
4	3.26±0.19	0.246±0.017	1.06±0.03	85.89±1.33	74.5±1.7	2.75±0.50	18.21±1.28	19.92	15.01
5	3.49±0.11	0.106±0.035	1.23±0.04	89.07±1.67	69.3±1.6	1.88±0.38	19.39±0.33	20.40	18.58
6	3.80±0.10	0.021± .007	1.30±0.08	97.75±1.20	71.2±2.5	0.0033±0.0006	23.03±1.08	25.16	21.10
7	2.98±0.08	0.165±0.018	1.30±0.04	92.38±1.88	74.3±2.8	4.60±0.50	20.10±0.26	20.58	19.67
8	3.82±0.10	0.35 ±0.006	1.31±0.05	97.00±0.30	76.2±4.0	0.0038±0.0013	22.13±1.25	23.25	19.12

TABLE 6

ULTIMATE TENSILE STRENGTH OF PRODUCTION PREPREG

Date	Lot. No.	No. Rolls Tested	Strand Tensile Strength (psi) Average	High	Low
April 1962	89/F911	13	337	359	298
April 1962	90/F911	13	342	353	319
May 1962	91/F917	15	338	349	306
May 1962	92/F923	26	346	372	310
May 1962	94/F935	13	362	400	329
June 1962	95/F942	15	400	419	370
June 1962	96/F944	15	396	405	376
June 1962	97/F950	15	373	388	335
July 1962	100/F961	15	383	406	328
July 1962	101/F965	15	382	405	353
July 1962	103/F973	15	378	402	352
July 1962	105/F971	15	374	399	353
July 1962	107/F979	14	376	414	360
August 1962	106/F976	15	381	396	352
August 1962	108/F983	15	367	390	339

TABLE 7

[illegible]	1	2	3	8	9	10	11	12	13	14	15	16	17
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible] (1)	0.5% [illegible]	[illegible]	0.5% [illegible]	55 ft/min	75 ft/min	[illegible] ft/min	390°F	390°F, 75 ft/min	340°F	80 ft/min	345°F	[illegible], 390°F	Same as [illegible]
[illegible], °F	389	397	354	365	382	382	390	391					
[illegible]						[illegible]							
0	1.721	1.753	1.796	1.923	1.827	1.704	-	1.809	1.781	1.754	1.793	1.786	1.801
1	-	-	-	2.000	1.905	1.769	-	1.930	1.787	-	-	-	1.811
2	-	-	-	2.095	1.917	-	-	1.967	-	-	-	-	-
3	1.844	1.898	1.843	2.203	1.934	1.855	-	-	1.891	-	-	-	1.903
4	1.880	1.995	2.020	-	-	1.913	2.295	2.084	1.907	1.930	1.848	1.853	2.019
5	1.904	2.002	2.094	2.258	1.987	1.933	2.381	2.111	2.015	1.849	1.842	1.831	2.092
6	1.942	2.037	2.136	2.259	2.082	1.960	-	2.128	2.037	1.917	1.934	1.870	2.151
7	2.005	2.066	2.194	2.313	2.121	1.983	2.390	2.146	2.050	1.945	1.955	1.902	2.184
8	2.725	2.239	2.354	2.333	2.134	2.065	2.393	2.173	2.099	1.965	1.968	1.936	2.253
9	2.768	2.381	2.433	2.354	2.159	-	2.507	2.184	-	-	-	-	-
10	2.806	2.439	2.475	2.388	2.181	2.055	2.520	-	-	2.019	2.049	1.945	-
11										2.049	2.053		
12										2.098	2.104		

(1) [illegible] parameter as indicated varied from the control conditions for each of the runs.

[illegible] Condition

[illegible] = 0.5% [illegible]
[illegible] = [illegible] ft/min
[illegible] = [illegible]°F
[illegible] at [illegible]°F = [illegible]

TABLE 8

GEL TIME WITH AGING AT ROOM TEMPERATURE

Run No.	1	2	3	8	9	10	13	14	15	16
Lot No.	F-1070	F-1070	F-1070	F-1102	F-1102	F-1118	F-1118	F-1154	F-1154	F-1155
Process Variable	.35 BDMA	Nominal	.75 BDMA	55 ft/min	75 ft/min	83 ft/min	340°F	80 ft/min	345°F	145 BDMA; 350°F
Aging Time, Days	GEL TIME, SEC									
0	193	142	108	106	119	177	167	176	166	170
1	202	151	118	101	131	-	-	164	152	184
2	171	134	119	101	117	-	-	170	154	160
3	179	150	90	-	-	-	-	182	169	169
4	157	133	96	-	-	119	109	155	155	167
5	155	134	89	107	127	119	107	165	134	158
6	152	138	96	101	130	120	101	164	145	147
7	-	129	102	109	118	118	97	161	155	165
8	155	130	78	103	125	-	-	148	142	152
9	-	-	-	97	118	-	-	151	148	145
10	-	-	-	-	-	116	92	158	136	155

TABLE 9

HORIZONTAL SHEAR STRENGTH WITH AGING AT ROOM TEMPERATURE

Run No.	1	2	3	8	9	10	11	12	13	14	15	16
Lot No.	F-1070	F-1070	F-1070	F-1102	F-1102	F-1118	F-1108	F-1108	F-1118	F-1154	F-1154	F-1155
Process Variable	0.35 PHR BDMA	Nominal (.55)	0.75 BDMA	55 ft/min	75 ft/min	[illegible] ft/min	390°F	390°F 75 ft/min	410°F	80 ft/min	345°F	.45 BDMA 350°F
Aging Time, Days	HORIZONTAL SHEAR STRENGTH (1), psi											
0	9770	11150	10440	11420	9420	9920	9440	11610	10000	8000	7820	9020
1	9210	10120	11050	-	11910	10820	-	11450	9550	8470	8450	9330
2	11320	10330	11580	11750	10260	-	11110	12040	-	-	-	-
3	9140	11550	11360	12100	12030	12410	11010	-	11860	9830	10260	8980
4	10840	11540	11570	-	-	11250	11280	12460	12120	9420	10640	9270
5	10080	11640	11800	11820	12130	12190	-	12620	12760	9670	9560	9970
6	10710	11010	11230	10610	12100	11590	-	12730	11790	10280	10920	10300
7	10150	11650	11870	11590	12030	11650	10360	10800	11580	10830	10650	11280
8	11250	11570	11450	11000	11450	11680	10490	10620	11580	10880	11230	10170
9	10880	11340	11340	10530	11310	-	9800	10850	-	-	-	-
10	11440	11280	11210	10130	10870	11550	6530	-	10850	10300	9590	8520
11										8270	4470	5240
12										7990	2980	3930

(1) Data represents an average of three (3) test specimens. All specimens were tested at room temperature after an initial 6 hour water boil test. The loading rate was based on a head-travel speed of 0.5 in/minute. For Run Nos. 1, 2 and 3 mandrel was heated to 150°F prior to and during winding of prepreg. No heat was applied to the remainder of the runs.

TABLE 10

NOL-RING COMPOSITE AND FIBER TENSILE STRESSES

WITH AGING AT ROOM TEMPERATURE

NOL Rin.[1] Tensile Strength, 10^3 psi

Run No.	1		2		3		8		9	
Lot No.	F-1070		F-1070		F-1070		F-1102		F-1102	
Process Variable	.35 HDMA		(Nominal)		.75 HDMA		55 ft/min		75 ft/min	
Aging Time, Days	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber
0	220.7	356.0	219.6	354.4	218.0	351.8	216.0	341.5	214.1	360.2
1	211.8	335.7	218.1	339.0	217.2	344.5	216.0	344.0	206.5	344.0
2	222.8	348.3	215.6	345.6	220.1	351.0	224.2	351.0	220.0	355.0
3	215.9	346.8	214.0	344.0	213.7	346.0	221.4	340.2	223.2	350.9
4	215.9	346.0	204.7	338.2	214.9	343.9	-	-	-	-
5	222.5	353.5	203.6	329.3	214.8	352.0	208.4	332.9	222.7	354.2
6	211.0	345.0	212.5	347.0	194.7	320.9	210.6	329.0	216.1	346.9
7	211.5	344.3	211.7	351.6	195.9	319.0	225.6	347.0	[illegible]	359.6
8	214.7	350.1	215.8	352.1	211.2	311.0	201.1	[illegible]	205.8	340.3
9	207.7	335.8	209.4	334.0	208.4	33.0	[illegible]	337.9	198.4	330.0
10	219.5	353.4	204.1	325.5	209.2	335.6	200.6	329.2	205.9	352.6
11										
12										

Run No.	10		13		14		15		16	
Lot No.	F-1118		F-1118		F-1154		F-1154		F-1155	
Process Variable	83 ft/min		340°F		80 ft/min		345°F		.045 HDMA	
Aging Time, Days	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber	Comp.	Fiber
0	195.	347.8	203.0	352.0	211.0	351.0	225.9	352.3	224.6	347.9
1	195.4	[illegible]	[illegible]	327.9	225.7	[illegible]	235.6	[illegible]	232.0	351.0
2	-	-	-	-	-	-	-	-	-	-
3	199.9	347.0	203.7	[illegible]	[illegible]	346.1	244.0	[illegible]	234.3	351.3
4	202.8	352.5	206.3	354.5	[illegible]	348.4	225.5	344.0	[illegible]	347.4
5	196.8	342.5	205.5	352.2	[illegible]	331.7	219.9	339.2	223.9	354.4
6	190.6	349.9	207.2	344.0	233.8	[illegible]	225.9	341.1	223.9	350.0
7	186.3	343.3	[illegible]	[illegible]	229.5	[illegible]	225.7	344.6	220.2	335.0
8	189.0	350.7	[illegible]	[illegible]	227.0	361.4	221.1	349.0	213.1	344.8
9	-	-	-	-	-	-	-	-		
10	190.3	335.3	[illegible]	[illegible]	[illegible]	[illegible]	211.9	[illegible]	[illegible]	320.5
11					191.6	[illegible]	[illegible]	[illegible]	[illegible]	319.2
12					[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

(1) NOL ring specimens were prepared with 23 turns of roving using 15-1[illegible] tension for a nominal composite thickness of 0.600 inch. For Run Nos. 1,2, and 3 [illegible] was heated to 150°F prior to and during winding. No heat was applied to the remainder of the runs. Data represents an average of three (3) test specimens.

TABLE 11

AEROROVE STRAND STRESS WITH AGING AT ROOM TEMPERATURE

Run No.	1	2	3	8	9
Lot No.	F-1070	F-1070	F-1070	F-1102	F-1102
Process Variable	.35 BDMA	Nominal	.75 BDMA	55 ft/min	75 ft/min
Aging Time, Days	AEROROVE STRAND TENSILE STRENGTH, 10^3 psi				
0	327.2	350.8	361.4	345.8	354.2
1	312.9	372.7	352.0	318.1	273.4
2	327.2	353.9	344.9	325.2	332.8
3	356.5	382.6	358.4	-	-
4	341.5	359.7	351.3	-	-
5	348.2	337.2	388.0	296.7	312.6
6	350.6	385.6	349.7	339.4	360.1
7	323.6	398.5	370.9	-	-
8	361.3	368.5	371.5	339.4	378.4
9	321.2	357.2	-	-	-
10	329.6	354.4	-	-	-

TABLE 12

VOLATILE CONTENT AT 180°, 250°, AND 325°F WITH AGING

Run No.	1			2			3		
Lot No.	F-1070			F-1070			F-1070		
Process Variable	.35 BDMA			.55 BDMA			.75 BDMA		
	VOLATILES,(1) %								
Aging Time, Days	180°F	250°F	325°F	180°F	250°F	325°F	180°F	250°F	325°F
0	.-	-	1.44	-	-	1.26	-	-	1.01
1	.52	1.66	2.03	.47	1.39	1.43	.43	1.05	1.07
2	.45	1.33	1.82	.42	1.25	1.60	.40	0.99	1.20
3	.59	1.44	1.68	.51	1.28	1.40	.43	0.83	0.97
4	.56	1.39	1.92	.43	1.23	1.67	.37	1.09	1.34
5	.55	1.33	2.03	.45	1.23	1.71	.45	1.04	1.32
6	.75	1.51	2.13	.73	1.48	1.95	.81	1.14	1.21
7	.44	1.59	2.00	.38	1.31	1.62	.45	1.48	1.70
8	.57	1.57	-	.39	1.44	-	.35	1.12	-

(1) Data represents an average of three (3) test specimens.

No test was performed for blank spaces indicated.

TABLE 13

A SUMMARY OF INCOMING QUALITY-CONTROL TEST DATA

A. Run No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
B. Lot No.	F-1070	F-1070	F-1070	F-1082	F-1082	F-1121	F-1102	F-1102	F-1102	F-1118	F-1108	F-1108	F-1118	F-1154	F-1154	F-1155
C. EDMA Content, PHR	.35	.55	.75	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.45
D. Special Gravity (Resin Bath)	.853	.853	.853	.863	.843	.833	.858	.853	.853	.853	.853	.853	.853	.837	.837	.837
E. Resin Bath Temp. °F	75	75	75	75	75	75	65	75	75	75	75	75	75	75	75	75
F. Running Speed, ft/min.	65	65	65	65	65	65	65	55	75	83	65	75	65	80	65	65
G. Tower Temp. (nominal), °F	360	360	360	360	360	360	360	360	360	360	390	390	340	360	345	350
H. Average Measured Temp, °F	363	357	354	367	364	363	359	365	368	361	390	391	347	368	345	350
I. Process Variable	C	(Nominal)	C	D	D	D	E	F	F	F	G	F G	G	G F	F G	C G
J. PREPREG PROPERTY																
1. Volatile, %	1.44	1.26	1.01	1.40	1.26	0.94	1.33	1.26	1.28	1.58	1.22	1.45	1.33	1.02	1.12	1.19
2. Resin Content, %	21.2	21.0	20.3	25.3	20.4	17.7	22.1	19.5	21.5	23.7	21.5	21.6	23.3	17.6	17.7	15.4
3. Resin Flow, %	10.3	10.0	8.9	13.3	8.1	4.4	10.2	7.2	9.6	12.6	10.2	10.3	12.0	7.1	8.3	6.5
4. Gel Time, Sec	193	144	110	134	126	154	144	105	120	177	125	162	167	145	140	201
5. Viscosity, Centistokes	1.736	1.776	1.780	1.835	1.865	-	1.872	1.972	1.812	1.704	1.968	1.881	1.758	1.775	1.774	1.781
6. Strand Weight, gm/yd.	.632	.632	.633	.634	.637	.644	.631	.632	.630	.629	.630	.636	.633	.642	.641	.647
7. Strand Stress, 10^3 psi	326.8	339.0	368.4	371.3	345.0	319.4	350.6	348.7	348.5	371.7	367.5	367.5	362.2	336.4	339.0	312.6
8. NOL Composite Stress, 10^3 psi	323.9	317.9	215.9	193.1	210.4	227.2	219.1	226.2	223.0	-	210.8	223.8	-	211.0	196.9	224.6
9. NOL Fiber Stress, 10^3 psi	356.0	352.5	347.0	330.4	336.0	336.6	348.5	348.2	358.9	-	328.6	363.9	-	351.4	352.3	347.9
10. Band Width (Lot), 10^{-3} in.	-	-	-	-	-	-	-	-	-	89	73	83	71	71	75	70
11. Band Width (Roll), 10^{-3} in.	77	67	66	71	68	-	-	69	71	-	-	-	-	-	-	-

TABLE 14

A SUMMARY OF OUTGOING QUALITY CONTROL TEST DATA

Run No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Lot No.	F-1070	F-1070	F-1070	F-1082	F-1082	F-1121	F-1102	F-1102	F-1102	F-1118	F-1108	F-1108	F-1118	F-1154	F-1154	F-1155
HEMA Content, phr	.35	.55	.75	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.55	.45
Virgin Gel Time, sec	222	163	128	158	158	153	164	164	164	154	155	155	154	167	167	240
Sol'n Gel Time	222	154	104	154	133	148	135	139	139	129	148	135	127	175	195	194
Sol'n Viscosity, Centistokes	.6263	.6329	.6254	.7252	.6267	.6501	.6415	.6303	.6351	.7052	.6400	.6700	.6987	.6000	.5975	.6192
Running Speed, ft/min	65	65	65	65	65	65	65	55	75	83	65	75	65	80	66	65
No. 1 Tower Temp. °F:																
Top	371	362	362	376	374	360	363	363	370	363	383	386	353	365	347	353
Gauge	360	357	353	362	360	358	359	359	360	360	380	380	340	368	345	355
Bottom	341	335	333	345	345	338	338	337	343	336	360	366	329	342	326	333
No. 2 Tower Temp. °F:																
Top	360	354	353	374	367	374	380	380	381	368	409	409	353	365	338	340
Gauge	360	353	352	356	353	359	360	360	362	354	390	390	340	372	352	350
Bottom	340	333	335	352	346	340	352	352	353	341	381	381	326	335	310	312
No. 3 Tower Temp. °F:																
Top	172	162	160	178	175	154	155	162	163	167	165	165	167	157	155	153
Gauge	165	152	143	161	161	139	140	149	158	156	150	150	150	147	145	140
Bottom	145	157	155	171	170	150	153	160	159	159	160	162	148	155	152	150
Prepreg Property																
1. Volatile, %	1.69	1.42	1.32	1.99	1.83	1.75	1.77	1.47	1.40	1.94	1.24	1.24	1.75	1.46	1.05	1.41
2. Resin Content, %	20.7	20.3	19.4	24.6	19.7	23.2	21.6	19.4	21.5	23.6	21.0	22.0	23.2	17.16	18.08	16.08
3. Resin Flow, %	7.40	8.03	7.23	11.6	7.35	11.7	10.2	6.7	10.7	12.5	8.12	10.6	11.2	6.48	6.95	5.97
4. Shrend Stress, 10^3 psi	321.9	343.8	344.8	-	-	-	-	-	-	-	348.5	355.8	-	-	-	-
5. Horizontal Shear, psi																
R.T./2-hr water boil	8280	8860	9640	10870	10950	9680	9560	9290	9550	9990	10420	10610	-	-	-	-
250°F/3min soak	1800	1680	1690	2150	1930	2300	1840	1380	1520	2300	1840	1600	-	-	-	-

TABLE 15

RESIN CONTENT WITH VARIOUS SPECIFIC GRAVITIES AND RUNNING SPEEDS

Run No.	Specific Gravity at 75°F	Running Speed, ft/min	Resin Content, %		
			No. of Rolls Tested	Av ($\bar{X}$)	Std Deviation
6	.833	65	4	17.66	0.31
15	.837	65	4	17.76	0.70
5	.843	65	6	20.33	0.75
1	.853	65	6	21.36	0.33
2	.853	65	6	21.06	0.57
3	.853	65	6	20.81	0.54
11	.853	65	6	21.80	0.51
12	.853	65	6	21.81	0.20
4	.863	65	6	25.33	0.63
8	.853	55	6	19.40	0.51
9	.853	75	6	21.48	0.22
10	.853	83	3	23.80	0.58
17(1)	.853	65	5	22.10	0.48
			5(3)	22.43	0.22
			5(3)	22.34	0.22
18(2)	.853	65	6	22.05	0.34

(1) Three rollers in resin bath. (2) Pre-wet roller. (3) Test in five areas within a roll.

TABLE 16

VISCOSITY AND GEL TIME WITH VARIOUS RUNNING SPEEDS

AND TOWER TEMPERATURES

Run No.(1)	Running Speed, (ft/min)	Tower Temp. °F	Viscosity, Centistoke/sec			Gel Time/sec		
			No. of Rolls Tested	Av ($\bar{X}$)	Std. Deviation	No. of Rolls Tested	Av ($\bar{X}$)	Std. Deviation
8	55	365	5	1.972	0.045	10	105	2
2	65	357	6	1.776	0.024	9	144	5
4	65	367	10	1.835	0.049	9	134	4
5	65	364	9	1.865	0.025	9	126	5
7	65	365	5	1.872	0.063	5	144	10
9	75	368	4	1.812	0.008	10	120	1
14	80	368	6	1.778	0.033	6	195	6
10	83	361	5	1.704	0.013	4	177	7
13	65	347	5	1.758	0.023	4	167	3
15	65	345	6	1.794	0.016	6	190	4
11	65	390	4	1.968	0.032	5	125	9
12	75	391	5	1.881	0.042	5	162	9

(1) Resin mixes used for these runs all contained 0.55 phr BDMA.

TABLE 17

GEL TIME AND VISCOSITY WITH VARIOUS AMOUNTS OF BDMA

Run No.	BDMA Content (phr)	Virgin Gel Time (sec)	Tower Temp. (°F)	Gel Time, Sec.			Viscosity, Centistoke/sec		
				No. of Rolls Tested	Av ($\bar{X}$)	Std. Deviation	No. of Rolls Tested	Av ($\bar{X}$)	Std. Deviation
1	.35	222	363	10	193	3	8	1.736	0.014
16	.45			6	200	3	6	1.791	0.014
2	.55	163	357	9	144	5	6	1.776	0.024
4	.55	158	367	9	134	4	10	1.835	0.049
5	.55	158	364	9	126	5	9	1.865	0.025
7	.55	164	365	5	144	10	5	1.872	0.063
3	.75	128	354	10	110	4	8	1.780	0.020

TABLE 18

TEST DATA ON RESIN CONTENT CONTROL RUNS

Run No.	17	18
Lot No.	F-118	F-1121
Control Device	3 rollers	Pre-wet roller
BDMA Content, phr	.55	.55
Specific Gravity (Resin Bath)	.853	.853
Resin Bath Temp. ^{o}F	75	75
Running Speed, ft/min	65	65
Tower Temp.	360	360
Prepreg Property		
1 Volatile, %	1.48	0.99
2 Resin Content (lot) %	22.1	22.0
3 Resin Content (roll) %	22.4	-
4 Resin Flow, %	10.6	9.3
5 Gel Time, sec	156	141
6 Viscosity, Centistokes	1.820	-
7 Strand Weight, gm/yd	.630	.639
8 Strand Stress, 10^3 psi	372.5	353.1

TABLE 19

RESIN CONTENT AND TENSILE STRENGTH

WITH VARIATIONS OF CREEL TENSION AND PACKAGE GEOMETRY

Run No.	Process Variable	Resin Content (%) with Creel Tension (lb)			Strand Tension, (10^3 psi)		NOL Ring Fiber Stress (10^3 psi)	
		1.5	2.0	4.0	60 Waywind	3 1/8 Waywind	60 Waywind	3 1/8 Waywind
1	.35 BDMA	21.8	21.4	20.9	319.0	325.6	362.5	353.6
2	.55 BDMA	21.5	21.4	20.3	355.9	351.1	349.0	353.7
3	.75 BDMA	20.2	20.8	20.1	340.4	366.3	342.0	346.3
4	.863 Sp. Gr.	27.3	25.3	24.0	358.8	377.6	329.6	326.1
5	.843 Sp. Gr.	21.2	20.3	19.9	330.8	347.8	331.4	336.8
6	.833 Sp. Gr.	17.4	17.7	18.4	331.2	318.1	333.9	338.0
7	65°F Bath Temp.	23.0	21.7	22.4	357.7	345.1	351.6	346.9
8	55 ft/min	19.0	19.4	19.8	329.4	347.3	356.3	340.0
9	75 ft/min	23.5	21.5	22.4	337.4	345.5	355.8	362.0
10	83 ft/min	25.0	23.8	23.4	375.0	368.7	-	-
11	390°F T. Temp.	25.2	21.8	21.0	372.0	364.7	330.2	327.0
12	390°F + 75 ft/min	21.6	21.8	21.4	347.3	369.0	366.0	361.8
13	340°F T. Temp.	24.6	23.4	23.0	386.9	380.3	-	-
14	80 ft/min + .837 Sp.Gr.	18.9	17.6	16.5	313.9	340.4	-	-
15	345°F T.Temp.; .837 Sp. Gr.	18.2	17.8	17.1	347.8	344.9	-	-
16	.45 BDMA; 350°F T.Temp.	17.0	15.1	15.3	316.2	310.7	-	-
17	R.C. control (3 rollers)	22.1	22.2	21.4	-	371.9	-	-
18	R.C. control (Pre-wet roller)	21.6	22.1	22.0	346.5	360.5	-	-

TABLE 20

[illegible] TENSION DURING [illegible] DETERMINATION

Run No. 8

Lot No. F-1102

Process Variable: [illegible] ft/min Running Speed

Roll No.		1	2	3	4	5	6	7	8	9	10
TENSION*, lb.											
1. Creel		2.0	2.0	2.0	2.0	2.0	2.0	1.5	1.5	4.0	4.0
2. Location A	(1)	4.5	4.5	5.0	4.5	5.0	5.0	4.0	5.0	5.5	5.0
	(2)	4.0	4.5	4.8	4.0	5.0	5.3	5.3	4.0	4.8	3.5
3. Location B	(1)	10.0	10.0	11.0	10.0	10.0	11.0	10.5	10.0	10.0	13.8
	(2)	9.0	10.0	9.8	9.0	7.5	9.0	9.0	6.8	10.0	10.0
4. Location C	(1)	17.0	17.0	11.0	15.0	17.5	17.0	18.0	14.0	14.0	10.0
	(2)	14.0	15.5	14.0	14.0	13.0	12.8	13.0	12.0	15.0	16.8
5. Location D	(1)	3.0	3.5	3.5	3.5	2.5	4.0	3.0	3.0	3.0	2.5
	(2)	3.3	3.0	2.8	3.0	2.5	2.5	3.0	3.0	2.5	3.5
RESIN CONTENT, %											
AGC Data		19.5	19.3	19.7	15.5	19.2	20.2	19.0	19.8	19.9	-
U.S.P. Data		19.6	19.1	19.1	17.8	18.8	20.6	18.8	19.8	20.6	-

Run No. 9
Lot No. F-1102

Process Variable: 75 ft/min Running Speed

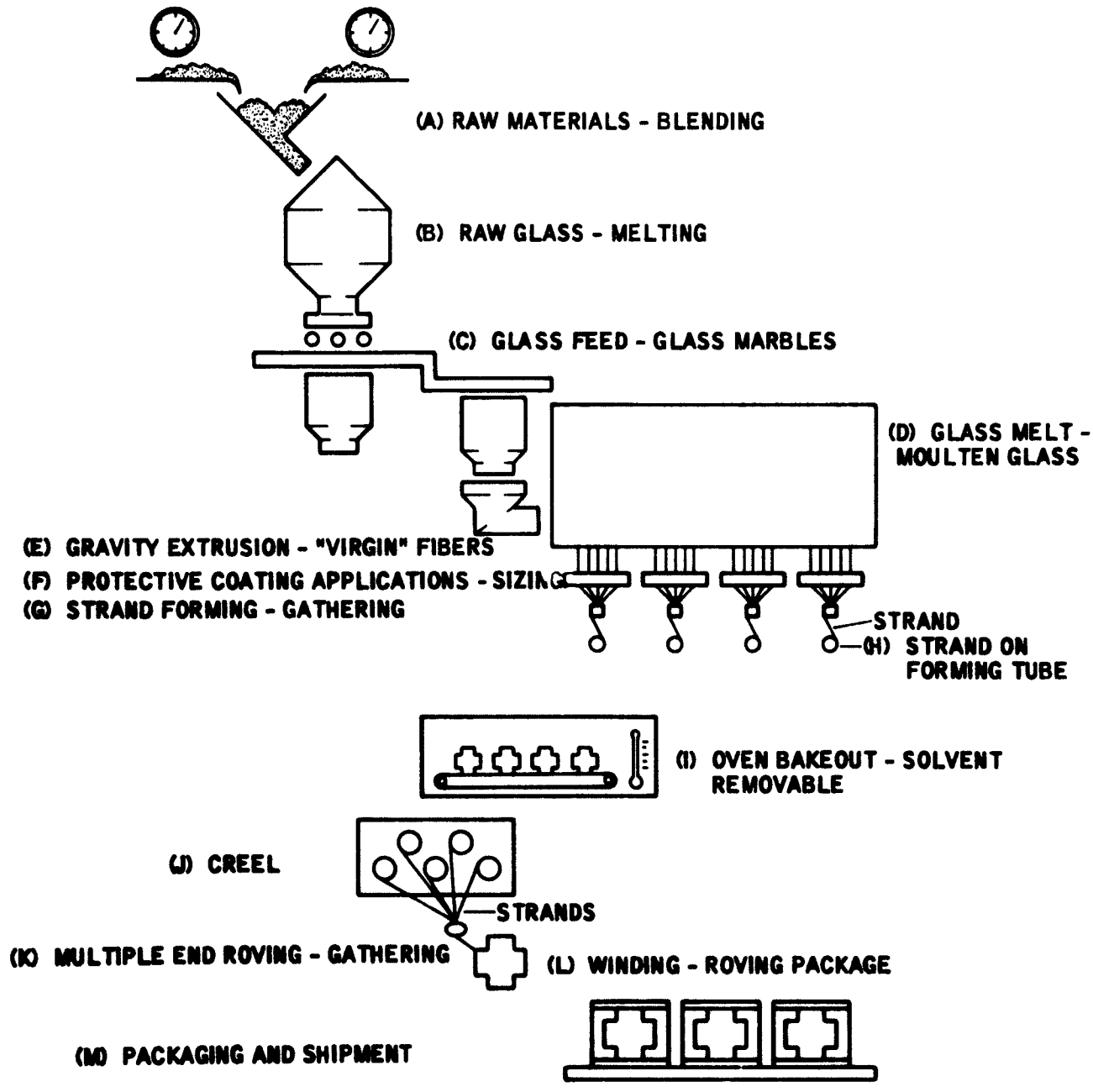
Roll No.	1	2	3	4	5	6	7	8	9	10
TENSION*, lb.										
1. Creel	2.0	2.0	2.0	2.0	2.0	2.0	1.5	1.5	4.0	4.0
2. Location A	5.0	4.5	4.5	3.5	4.8	5.0	3.5	4.5	4.5	4.5
3. Location B	10.0	10.0	10.5	15.5	14.8	13.5	15.0	17.0	17.0	14.5
4. Location C	15.0	13.0	11.0	11.5	11.5	17.0	17.5	18.5	17.0	17.0
5. Location D	5.0	1.5	5.0	5.0	5.0	4.5	4.5	4.8	4.8	5.0
RESIN CONTENT, %										
AGC Data	21.4	21.6	21.6	21.2	21.3	21.8	23.2	23.9	23.0	21.7
U.S.P. Data	22.2	21.6	21.6	21.2	21.9	22.2	18.7	22.0	21.8	21.9

*Location of measurements is shown in Figure 2.

TABLE 21

BAND-WIDTH MEASUREMENTS

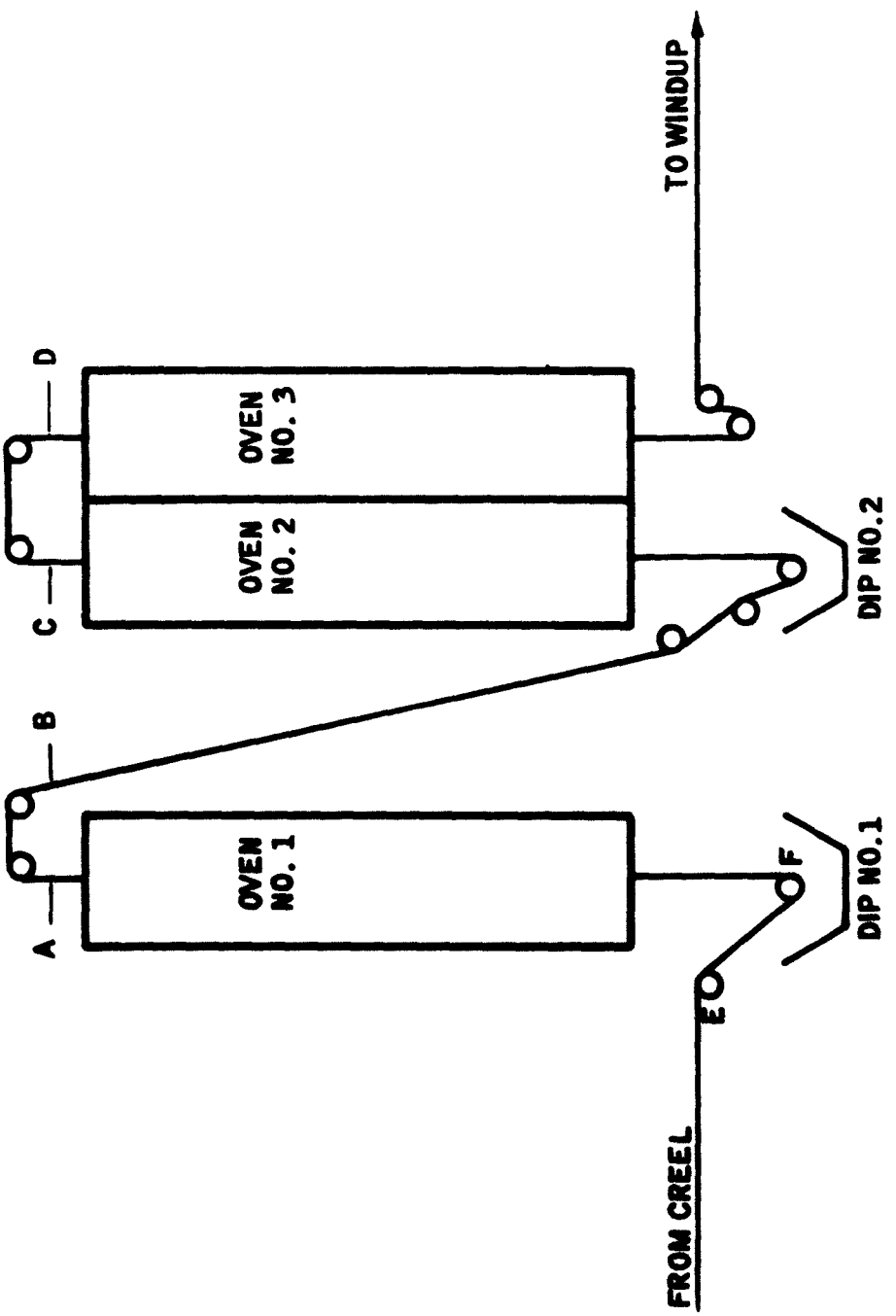
Run No.	Lot No.	Process Variable	Band Width, 10^{-3} inch				
			No. Tension	10 lb Weight Tension			
				Loc. 1	Loc. 2	Loc. 3	Edge
1	F-1070	0.35 BDMA	85	77	73	73	106
2	F-1070	Nominal (0.55)	76	76	67	67	92
3	F-1070	0.75 BDMA	76	66	65	66	118
4	F-1082	0.863 sp. gr.	77	76	70	67	96
5	F-1082	0.843 sp. gr.	72	71	70	64	80
8	F-1102	55 ft/min.	74	64	71	72	97
9	F-1102	75 ft/min	76	76	69	68	97
10	F-1118	83 ft/min	100	98	89	81	116
11	F-1108	390°F	74	71	73	76	98
12	F-1108	390°F + 75 ft/min	84	79	80	91	101
13	F-1118	340°F ft/min	82	72	69	72	102
14	F-1154	80 ft/min	79	71	70	70	103
15	F-1154	345°F	84	70	76	79	109
16	F-1155	0.45 BDMA + 350°F	73	68	72	71	93
18	F-1121	R.C. Control	76	78	72	75	100



FIBERGLASS MANUFACTURING PROCESS

A32/:63-968

Figure 1

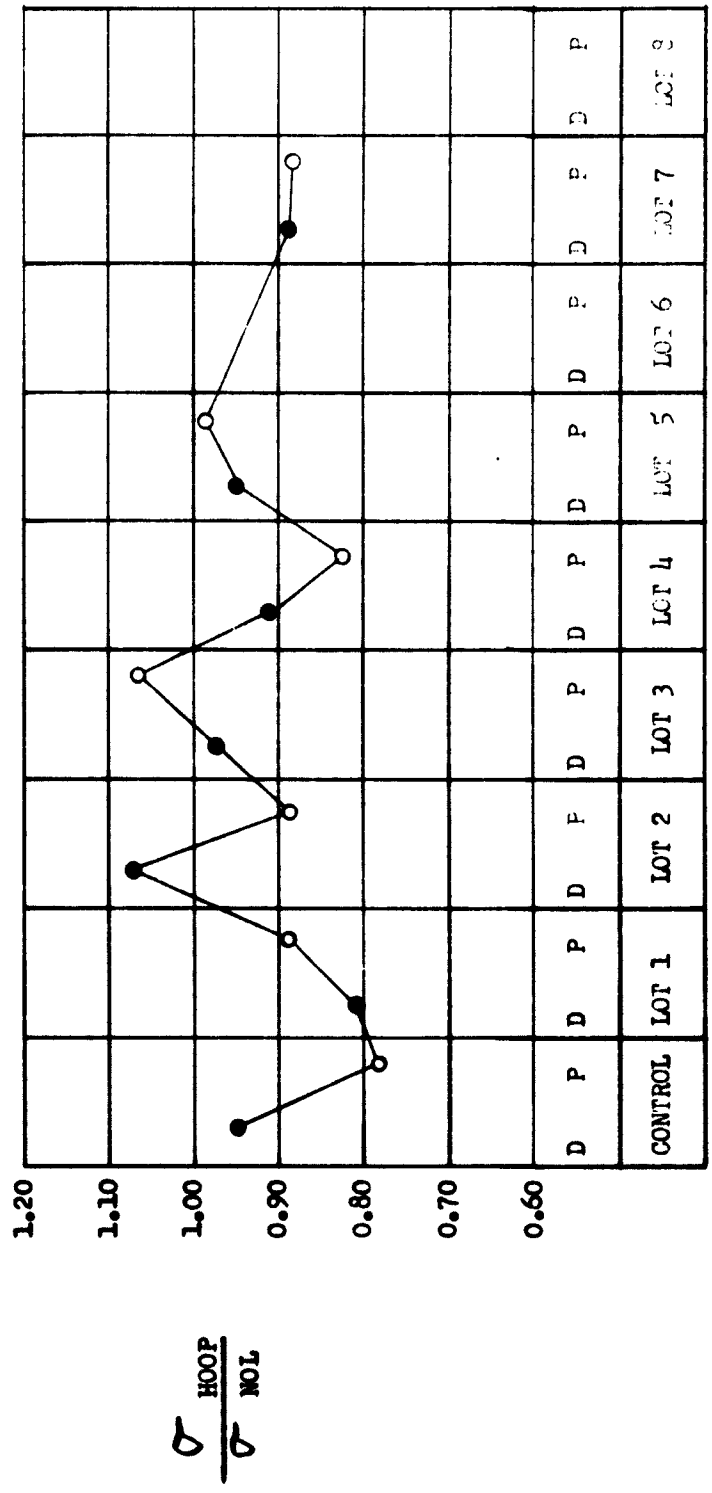


SCHEMATIC DIAGRAM OF U.S. POLYMERIC PREIMPREGNATION LINE

A270:63-903

Figure 2

COMPARISON OF NOL-RING DATA AND CHAMBER DATA



$\frac{\sigma_{HOOP}}{\sigma_{NOL}}$

Figure 3

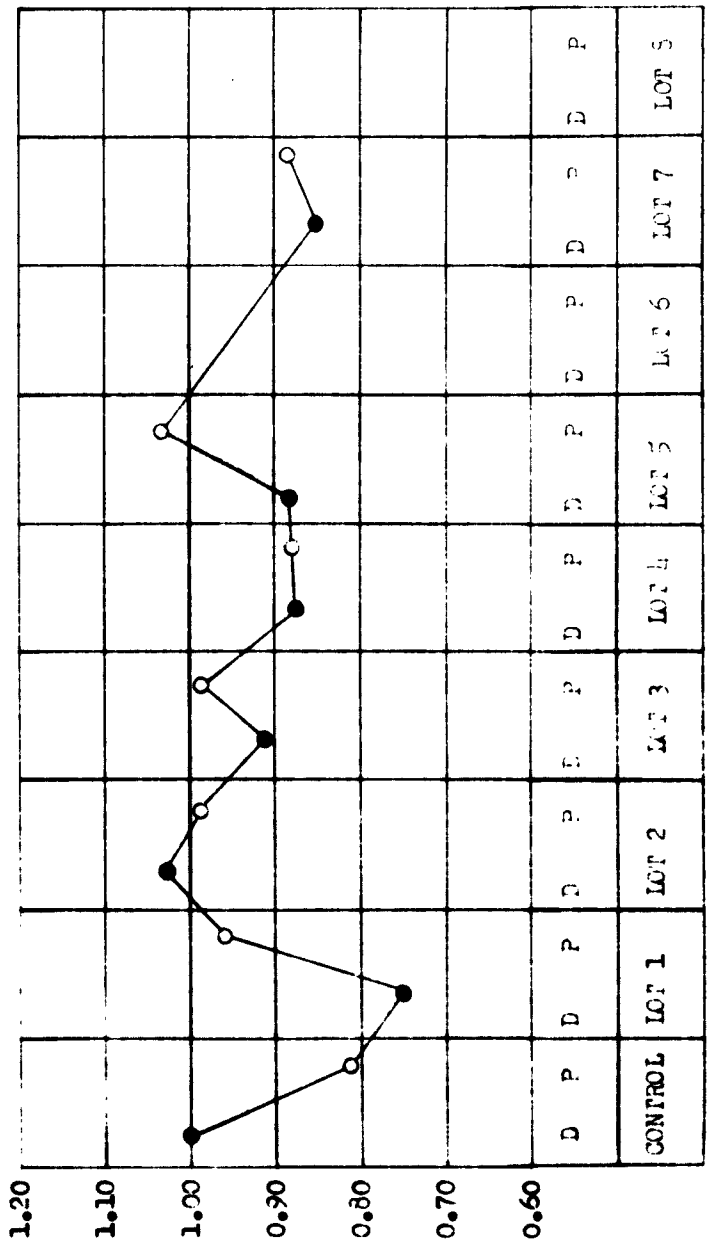

1.20
1.10
1.00
0.90
0.80
0.70
0.60
D P
CONTROL
D P
LOT 1
D P
LOT 2
D P
LOT 3
D P
LOT 4
D P
LOT 5
D P
LOT 6
D P
LOT 7
D P
LOT 8
$\frac{\sigma_{HOOP}}{\sigma_{STRAND}}$

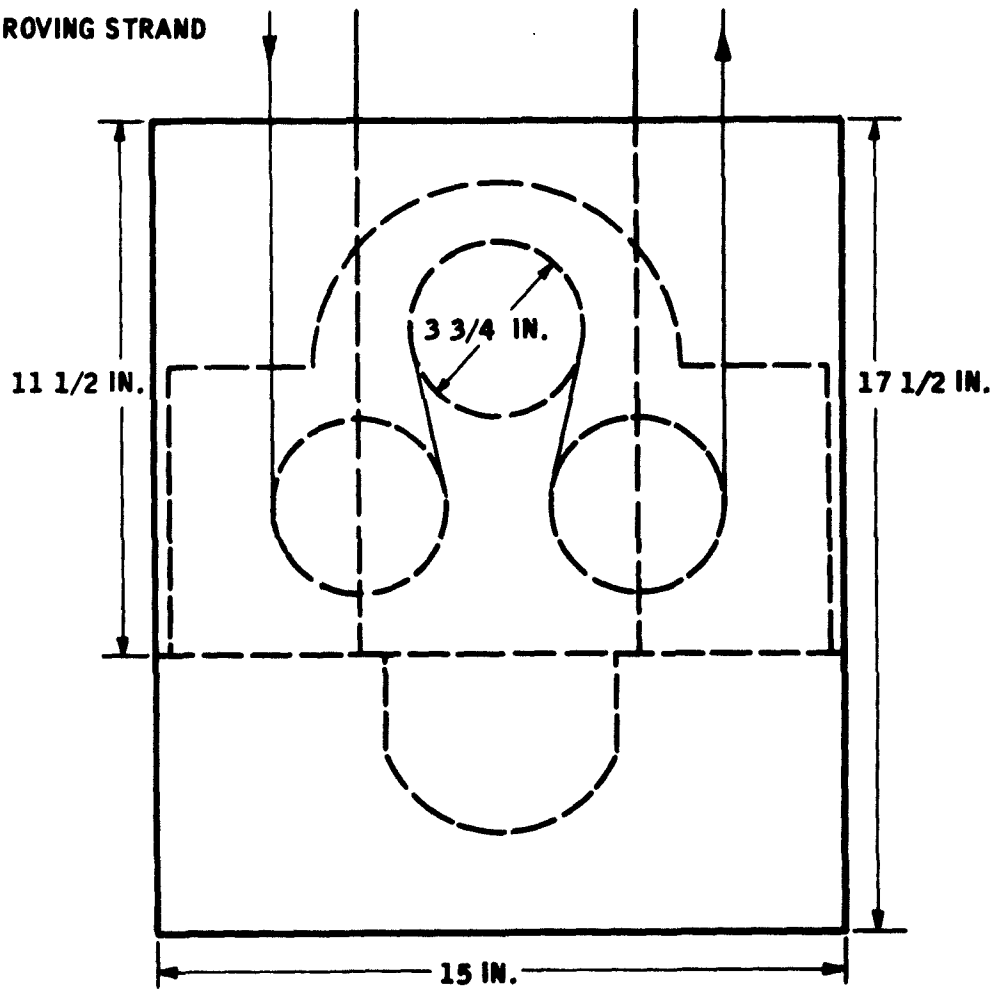
Figure 4

EQUIPMENT:

THREE 302/304 STAINLESS STEEL ROLLERS. LENGTH - 42 IN. DIAMETER 3 3/4 IN.

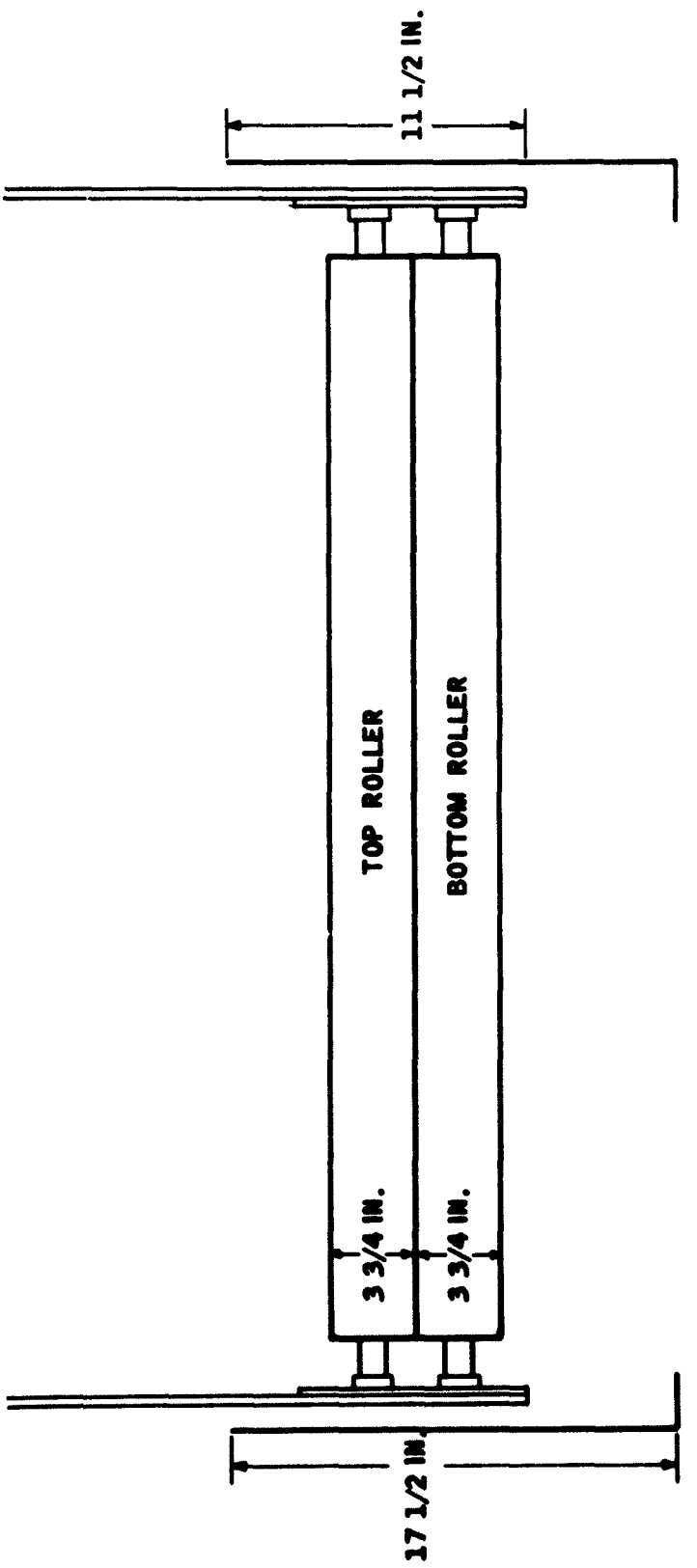
SIX 302/304 STAINLESS STEEL END CAPS FOR ROLLERS

SIX BRONZE BUSHINGS TO FIT END CAPS



SKETCH FOR ROLLERS IN RESIN BATH
(END VIEW)

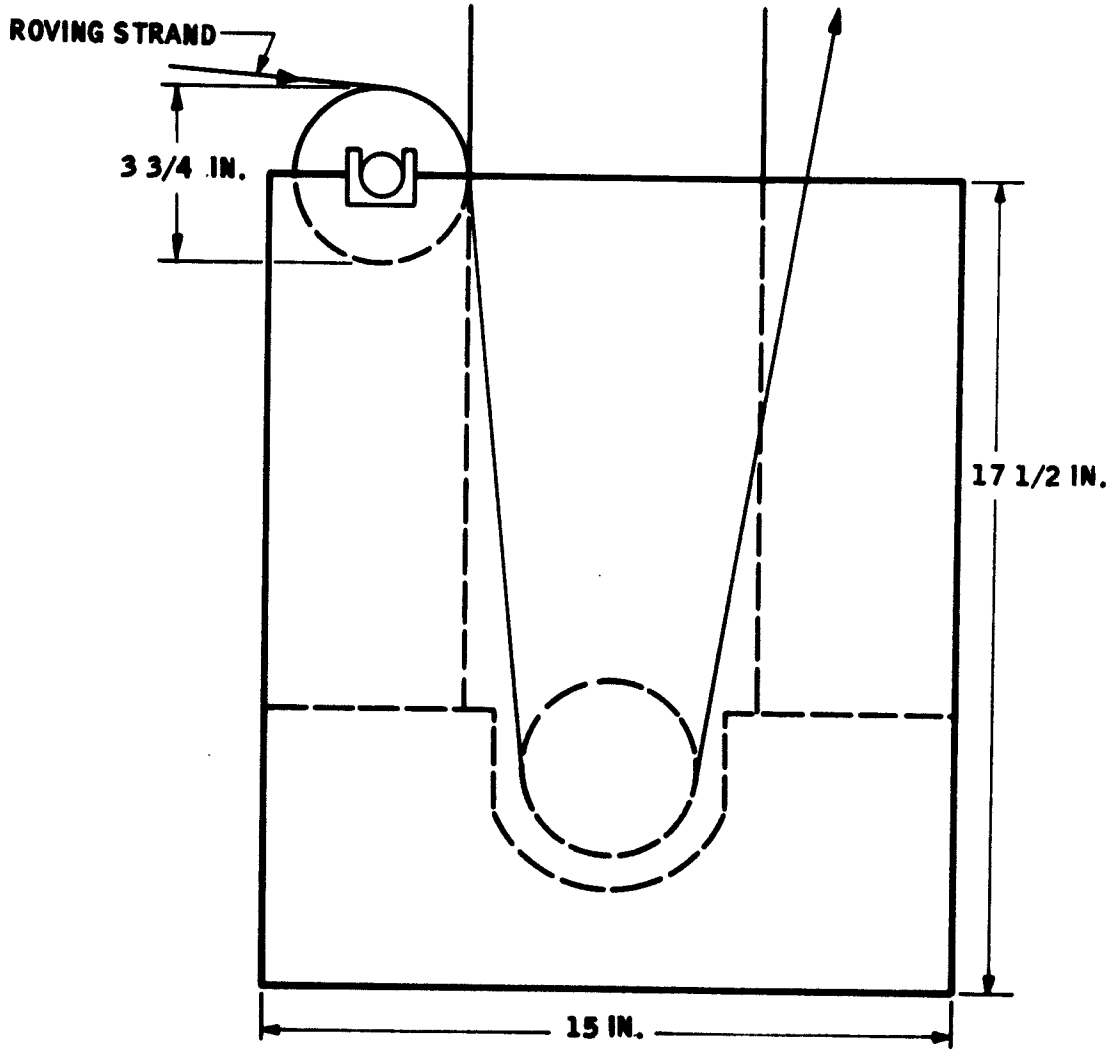
Figure 5



ROLLERS SHOWN WITH PAN SIDES AS REFERENCE
(SIDE VIEW)

A270:63-855

Figure 6



PAN WITH PRE-WET ROLLER
(END VIEW)

A270:63-857

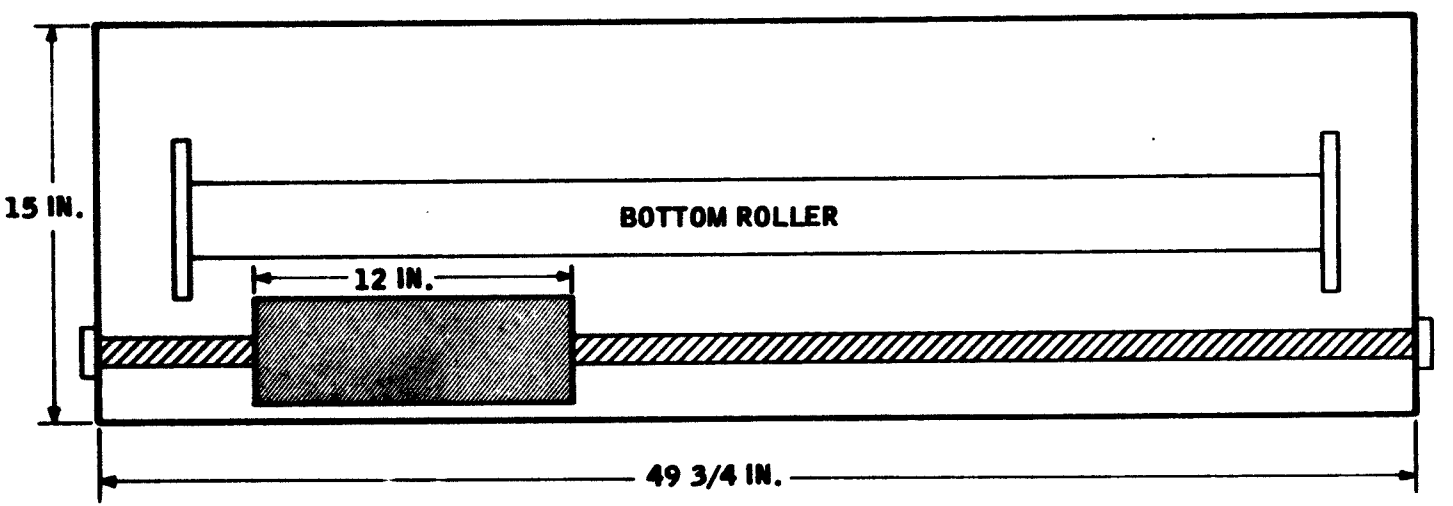
Figure 7

EQUIPMENT:
ONE ONE-FOOT 302/304 STAINLESS STEEL ROLLER, DIAMETER: 3 3/4 IN.

TWO 302/304 STAINLESS STEEL END CAPS FOR ROLLER

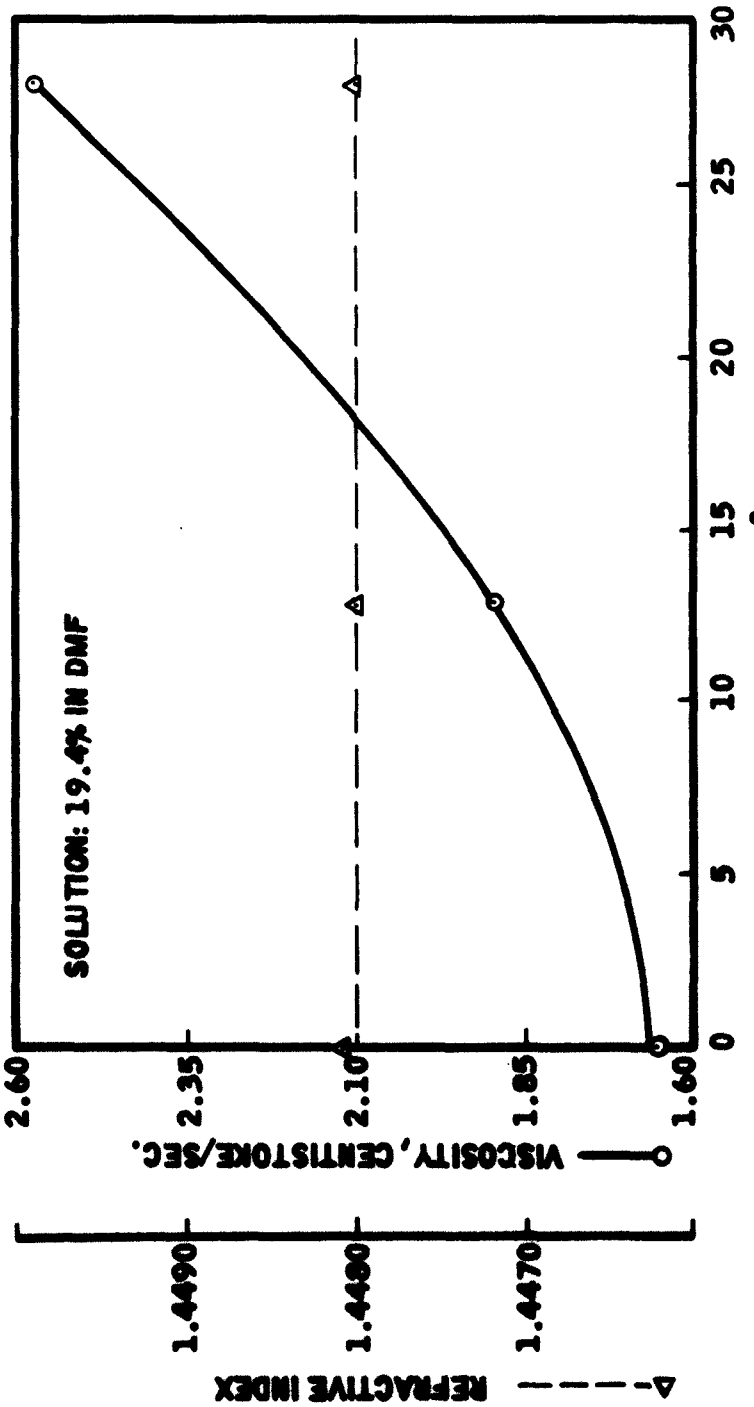
ONE FOUR-FOOT 302/304 STAINLESS STEEL SHAFT. DIAMETER 1 1/2 IN.

TWO TAILOR-MADE BRONZE BUSHINGS WITH SUPPORTS FOR BUSHINGS TO FIT THE ENDS OF THE SHAFT FOR ROTATION



PAN WITH PRE-WET ROLLER
(TOP VIEW)

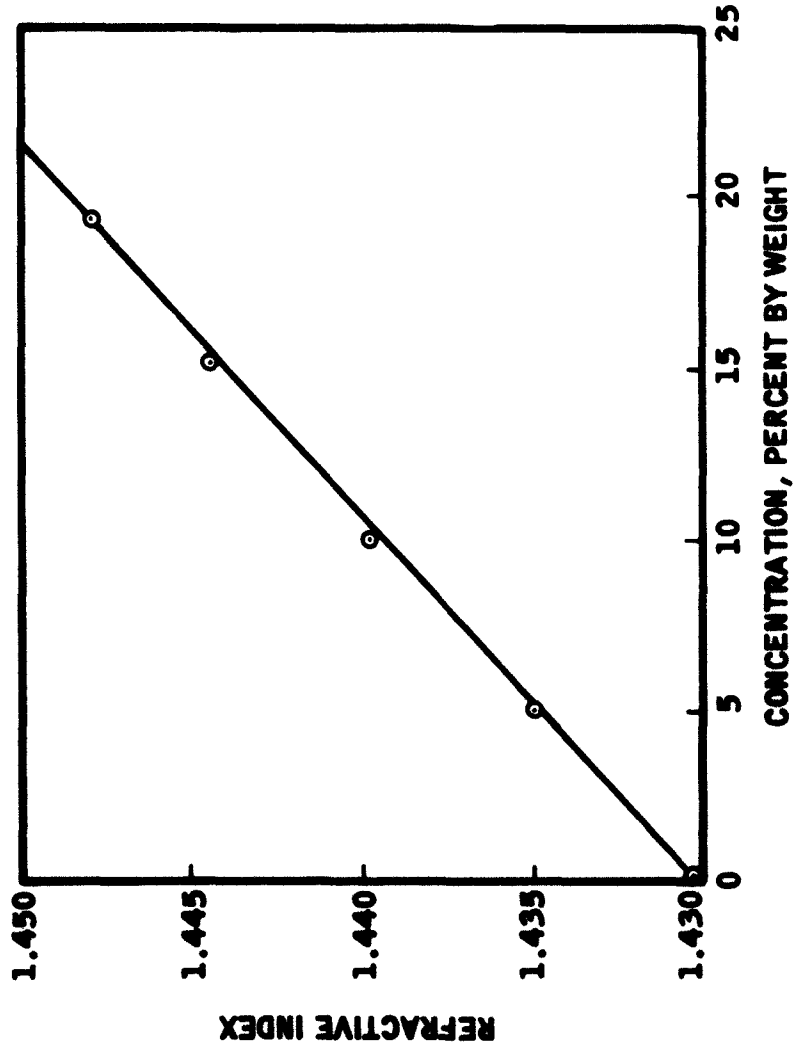
Figure 8



EFFECT OF TIME AT 215°F ON VISCOSITY OF 58-68R/0.55 BDMA RESIN SYSTEM

A270:63-852

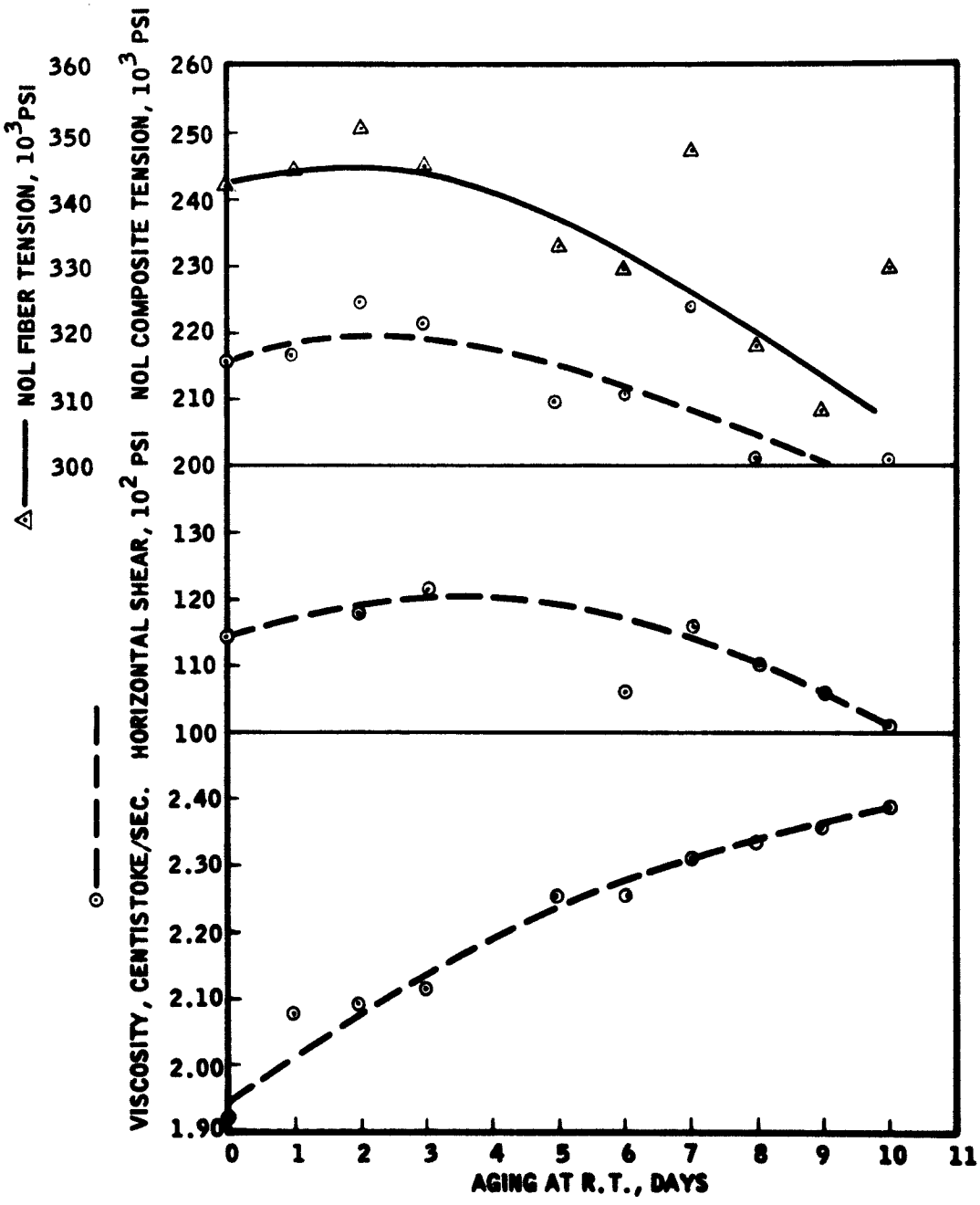
Figure 9



REFRACTIVE INDEX AS A FUNCTION OF CONCENTRATION OF 58-68R/0.55 BDMA RESIN SYSTEM IN DMF SOLVENT

A270:63-851

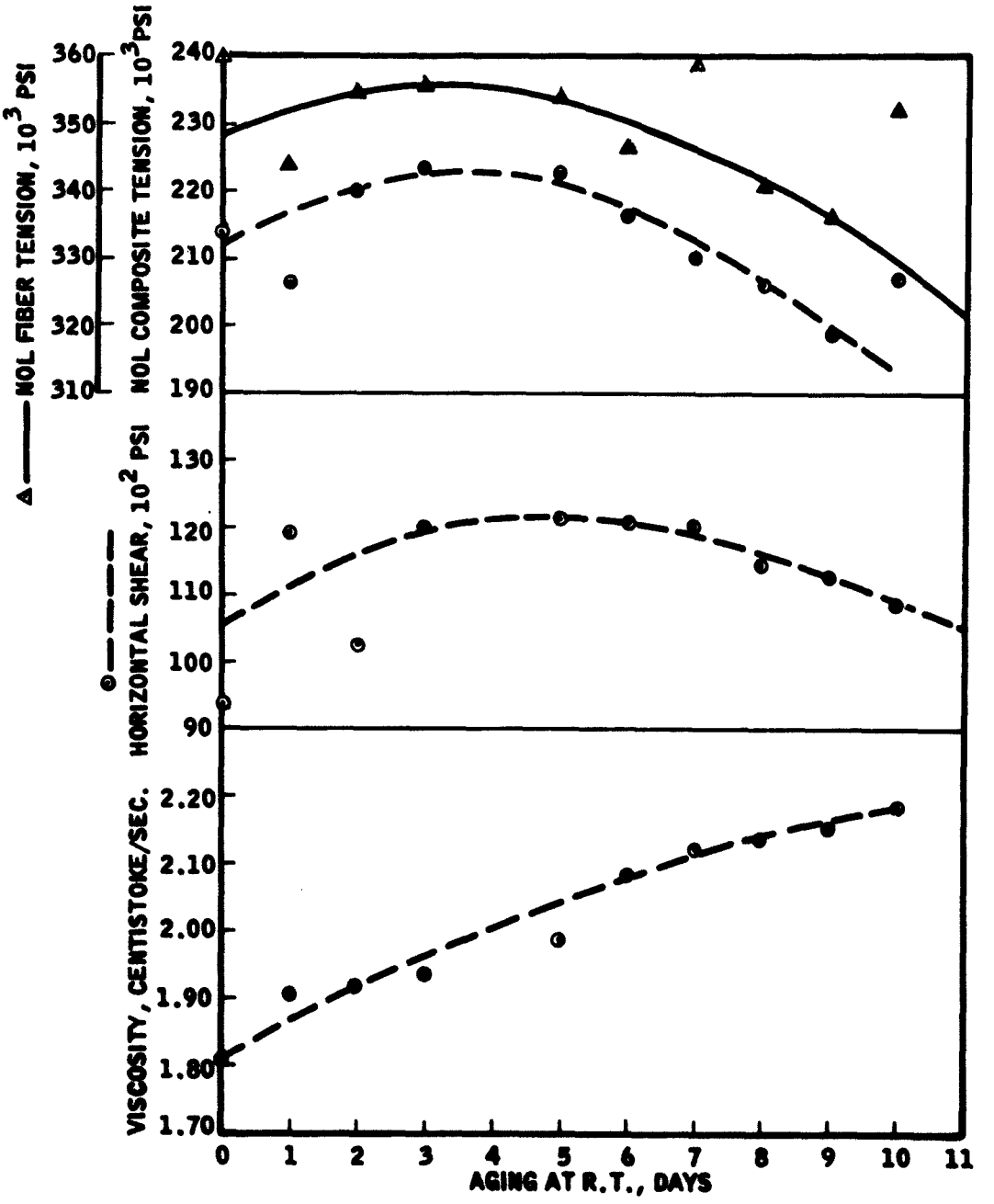
Figure 10



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: RUNNING SPEED AT 55 FT/MIN.)

A270:63-875

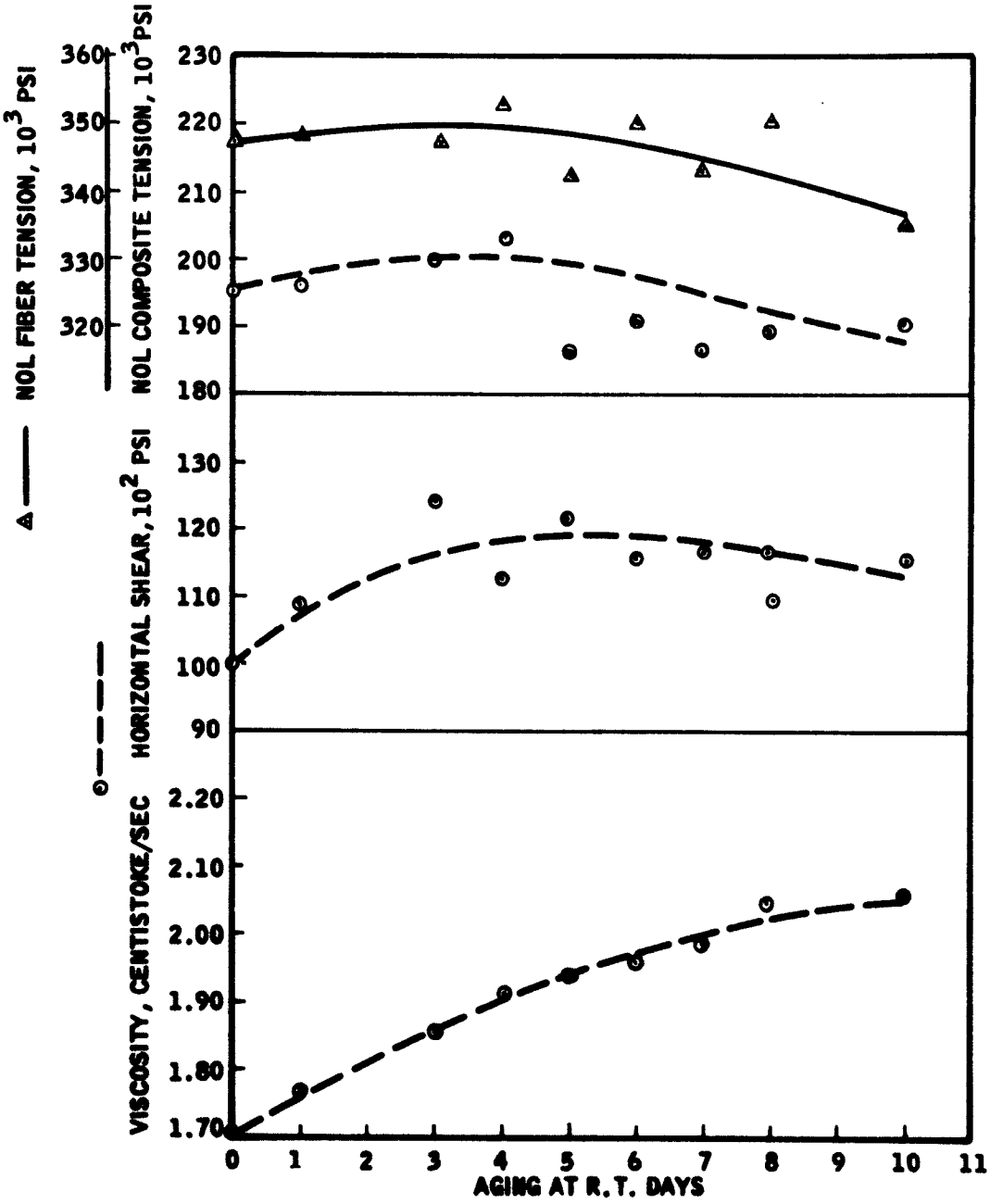
Figure 11



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: RUNNING SPEED AT 75 FT/MIN.)

A270:63-873

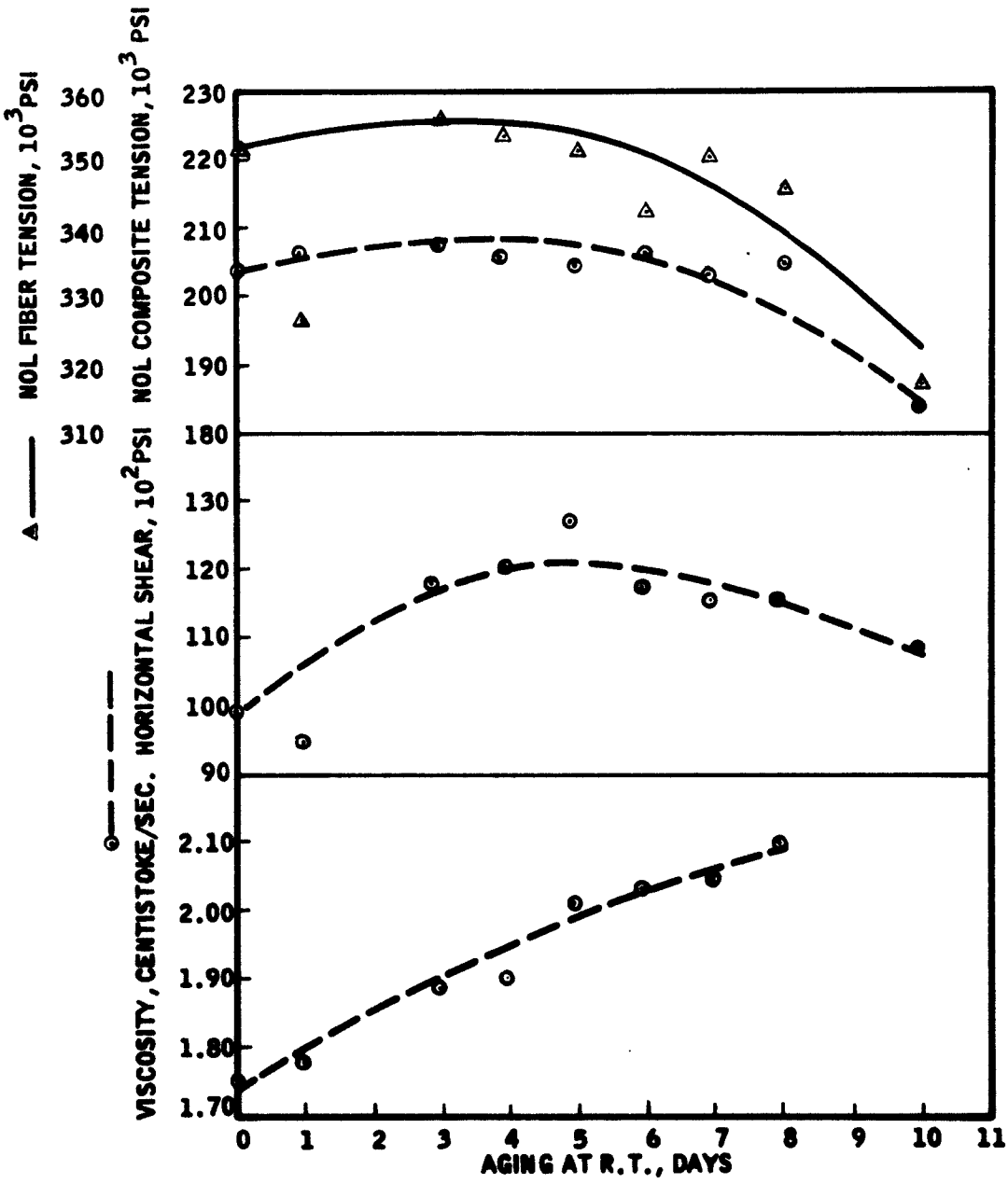
Figure 12



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: RUNNING SPEED AT 83 FT/MIN.)

A270:63-876

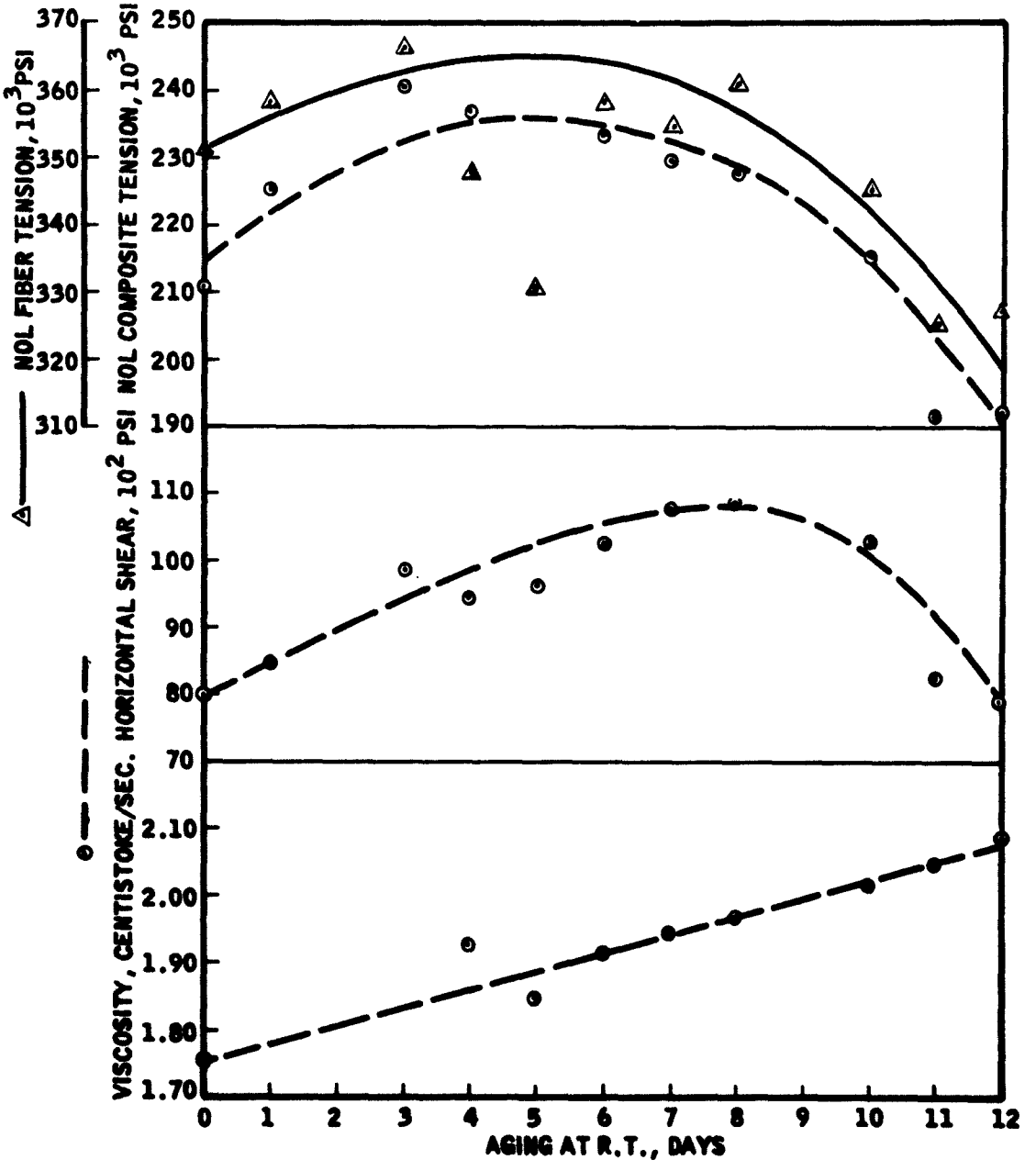
Figure 13



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: TOWER TEMPERATURE AT 340°F)

A270:63-877

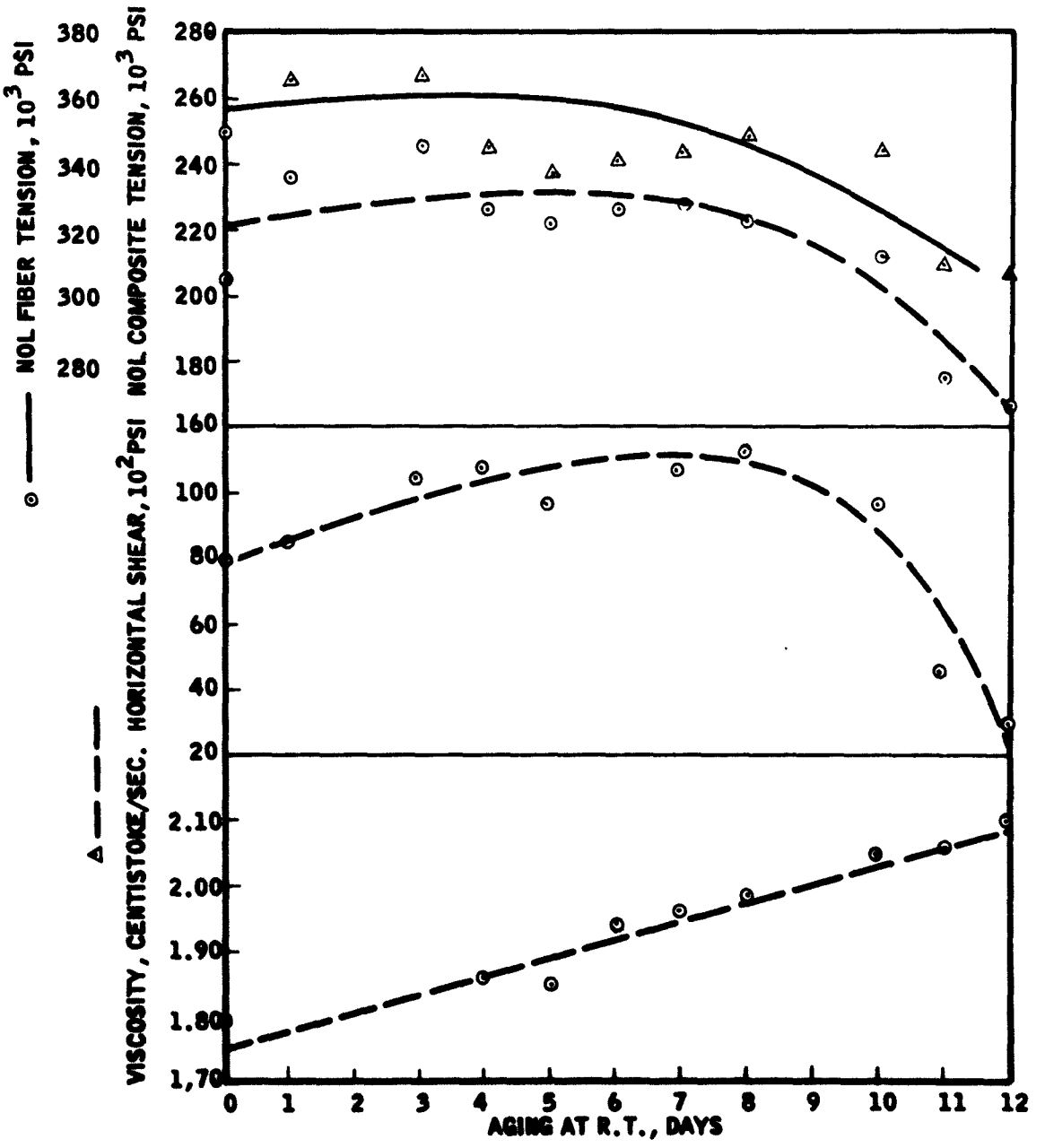
Figure 14



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: RUNNING SPEED AT 80 FT/MIN)

A270:63-873

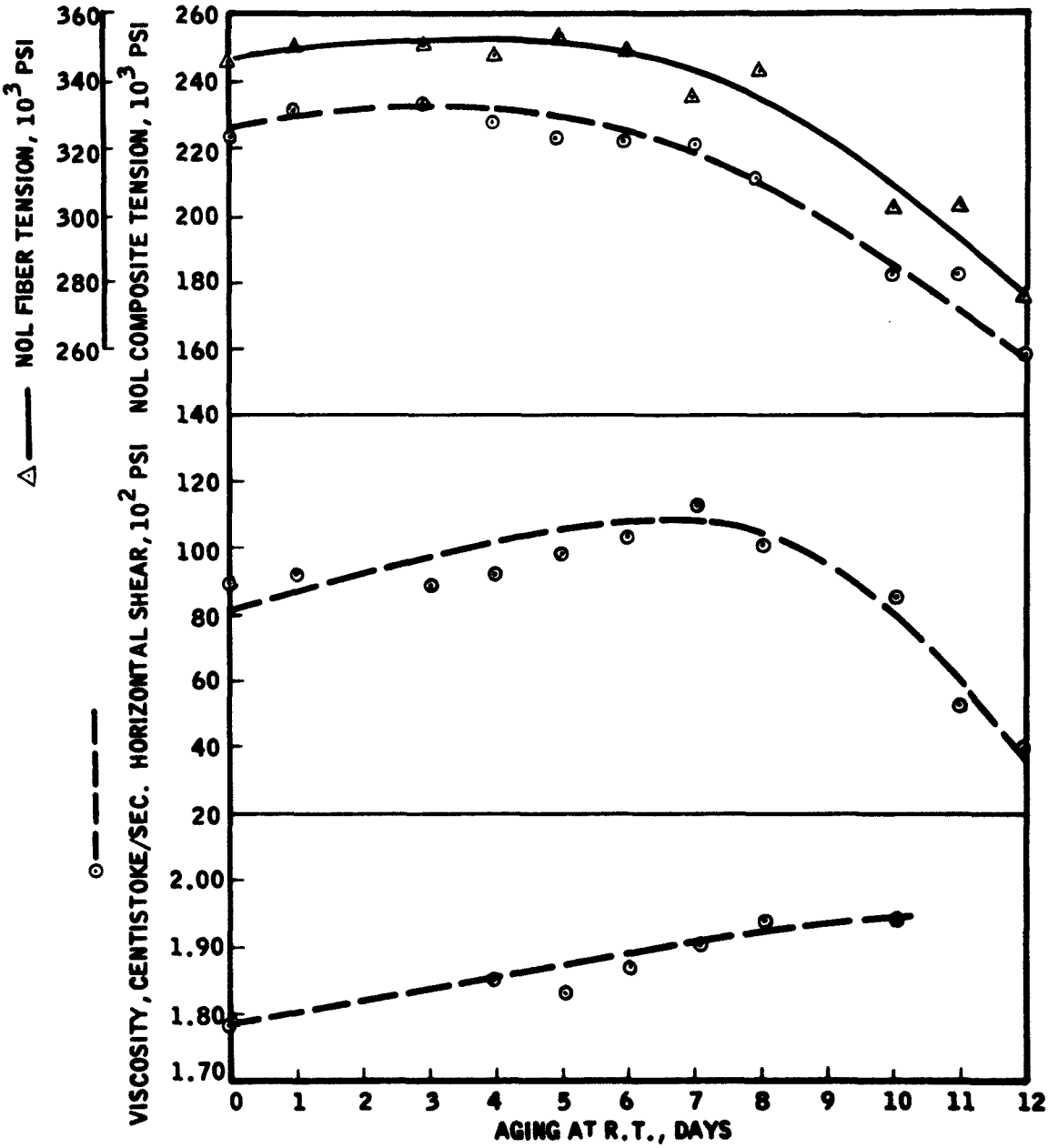
Figure 15



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE: TOWER TEMPERATURE AT 345°F)

A270:63-878

Figure 16



CORRELATION OF HORIZONTAL SHEAR AND NOL-RING TENSILE STRENGTHS WITH VISCOSITY UPON AGING (PROCESS VARIABLE = 0.45 BDMA CONTENT)

A270:63-885

Figure 17

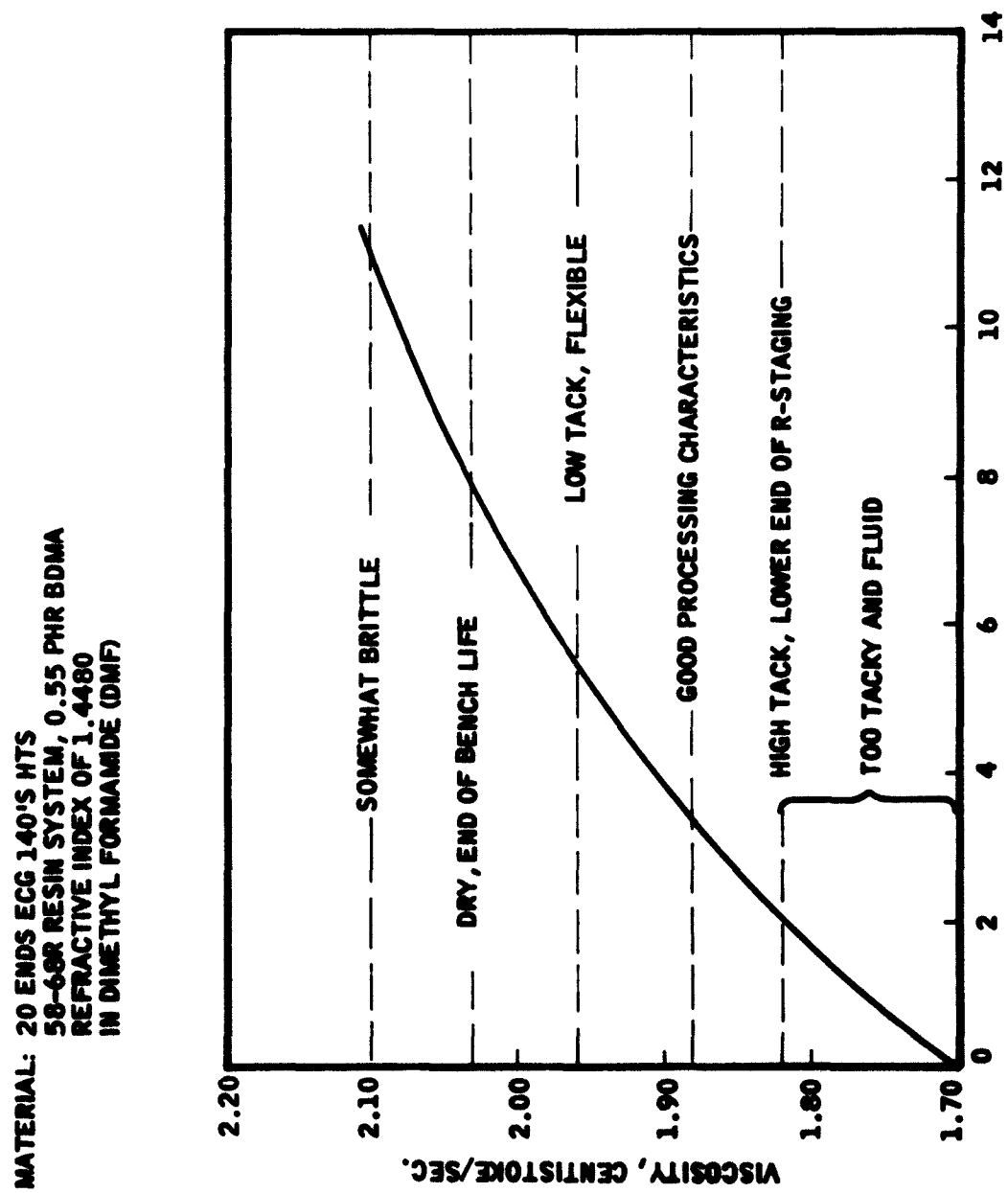
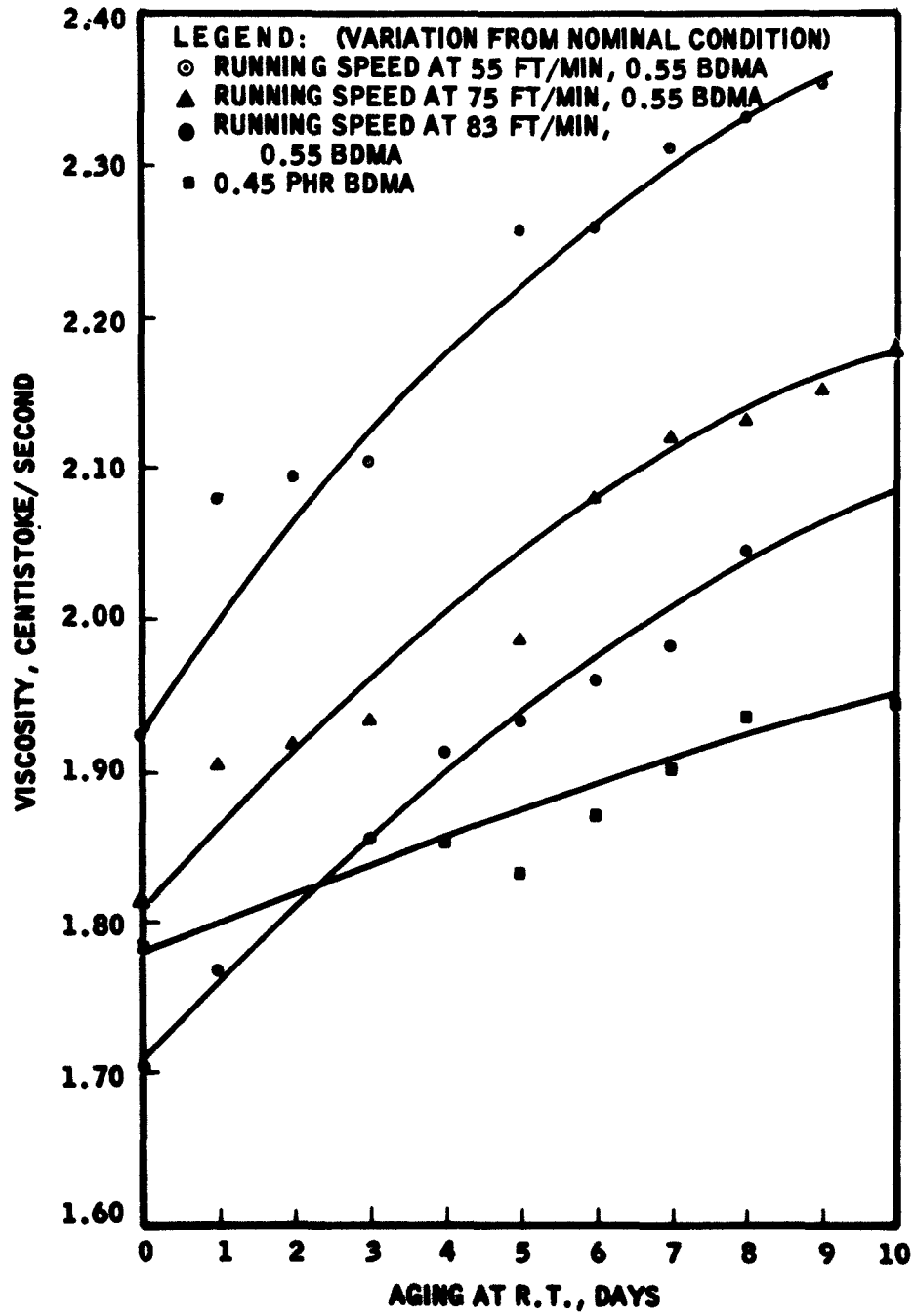

MATERIAL: 20 ENDS ECG 140'S HTS
58-60R RESIN SYSTEM, 0.55 PHR BDMA
REFRACTIVE INDEX OF 1.4480
IN DIMETHYL FORMAMIDE (DMF)
VISCOSITY, CENTISTOKE/SEC.
2.20
2.10
2.00
1.90
1.80
1.70
SOMEWHAT BRITTLE
DRY, END OF BENCH LIFE
LOW TACK, FLEXIBLE
GOOD PROCESSING CHARACTERISTICS
HIGH TACK, LOWER END OF R-STAGING
TOO TACKY AND FLUID
0
2
4
6
8
10
12
14
AGING AT R.T. (75°F), DAYS
EFFECT OF AGING ON VISCOSITY
A270:63-850

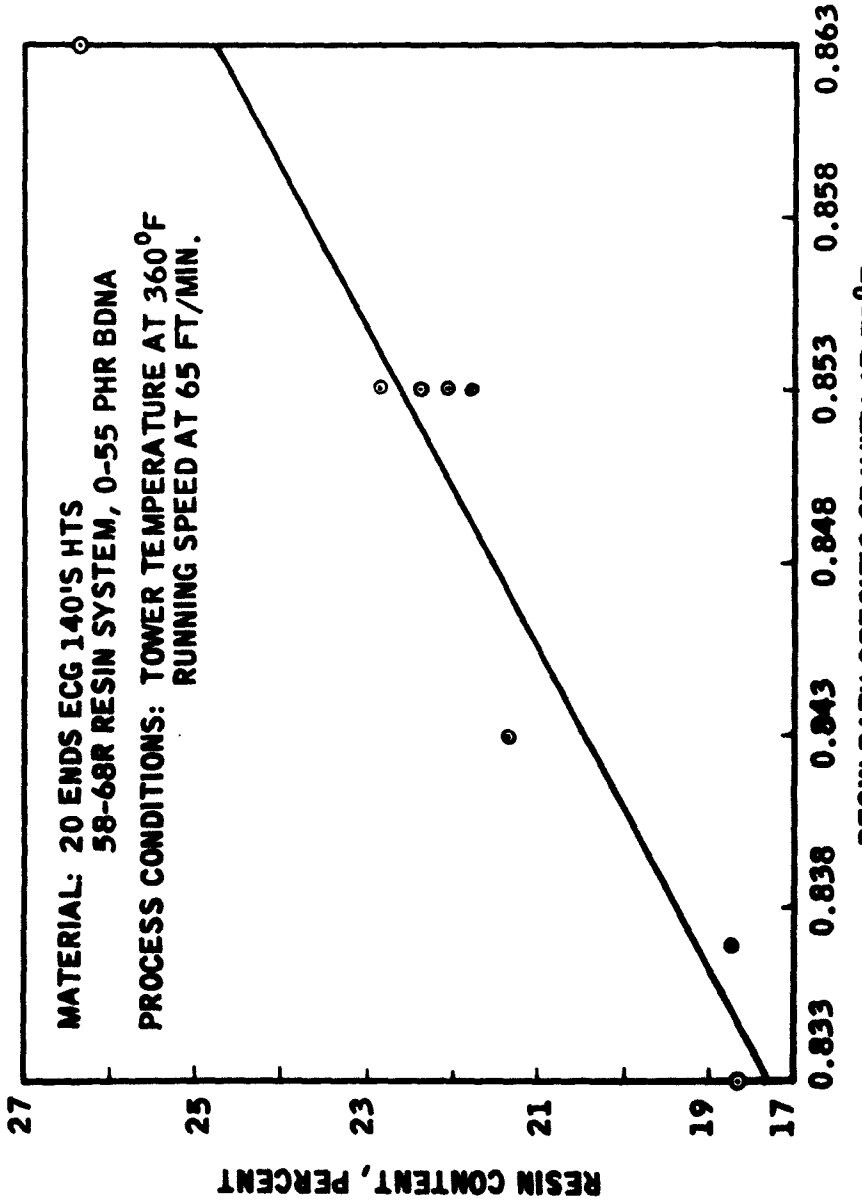
Figure 18



EFFECT OF AGING ON VISCOSITY FOR PREPREGS PRODUCED UNDER VARIOUS PROCESSING CONDITIONS

A270:63-884

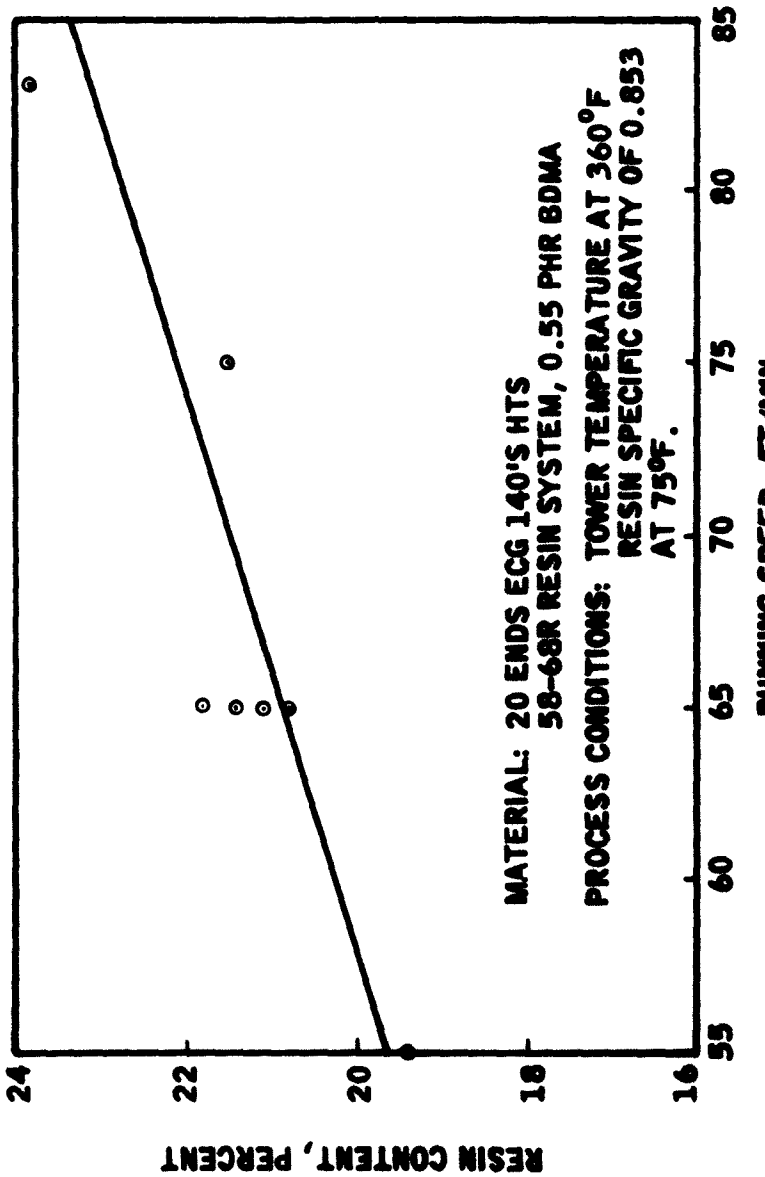
Figure 19



EFFECT OF SPECIFIC GRAVITY OF THE RESIN IMPREGNATING BATH UPON RESIN CONTENT

A270:63-879

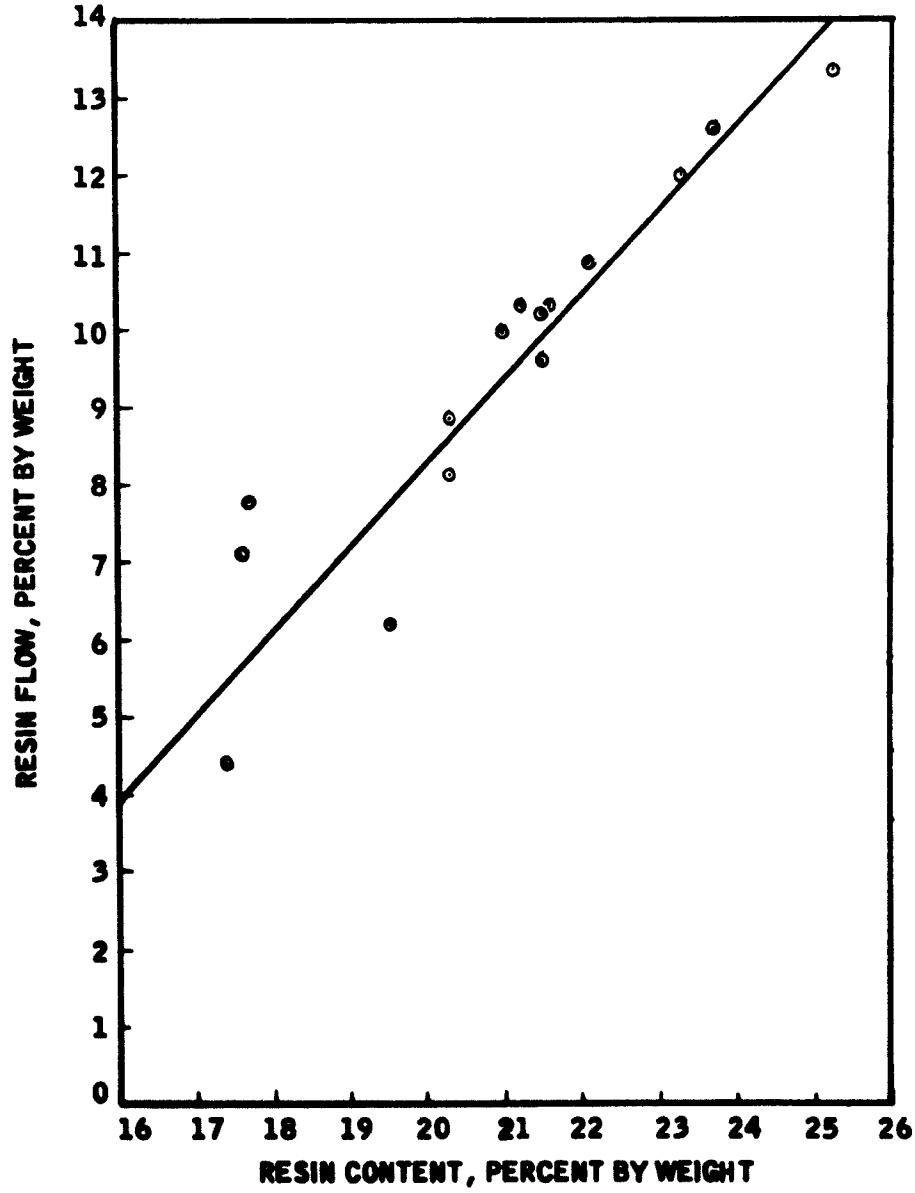
Figure 20



EFFECT OF RUNNING SPEED ON RESIN CONTENT

A270:63-880

Figure 21



CORRELATION OF RESIN CONTENT AND RESIN FLOW

A270:63-883

Figure 22

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: RUNNING SPEED AT 65 FT/MIN.
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F

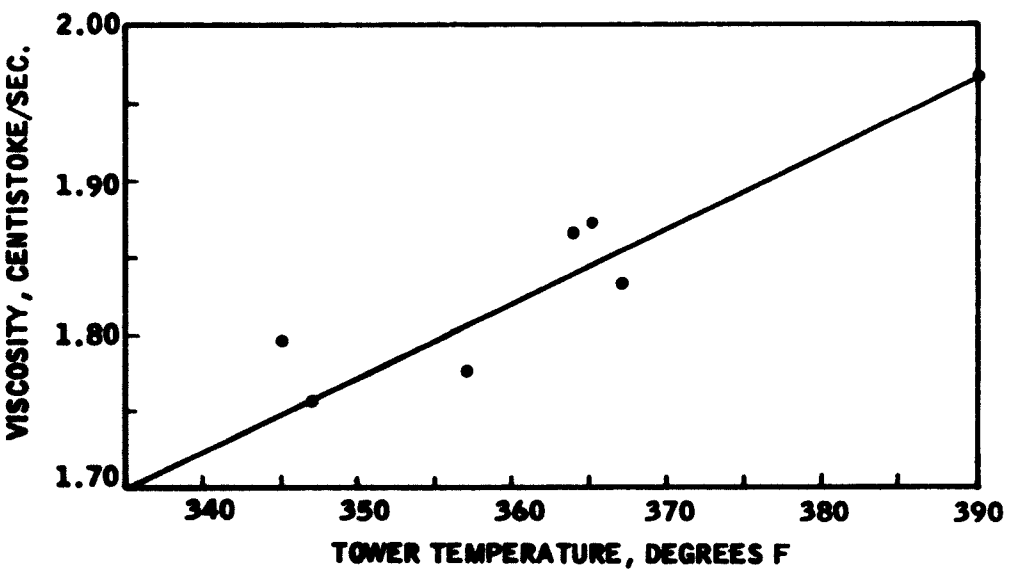
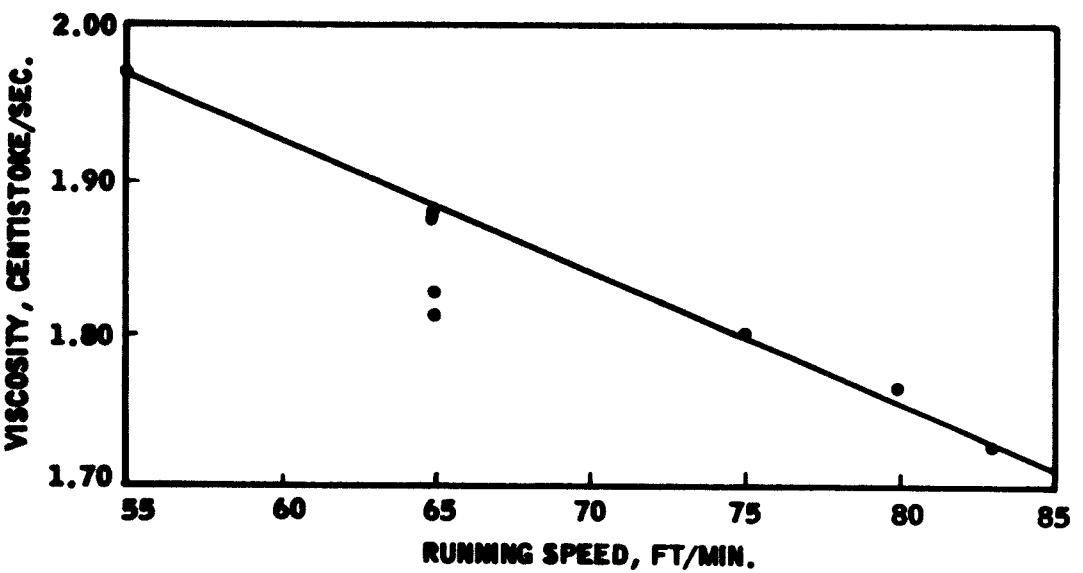


Figure 23

EFFECT OF TOWER TEMPERATURE ON VISCOSITY

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: TOWER TEMPERATURE AT 365°F
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F

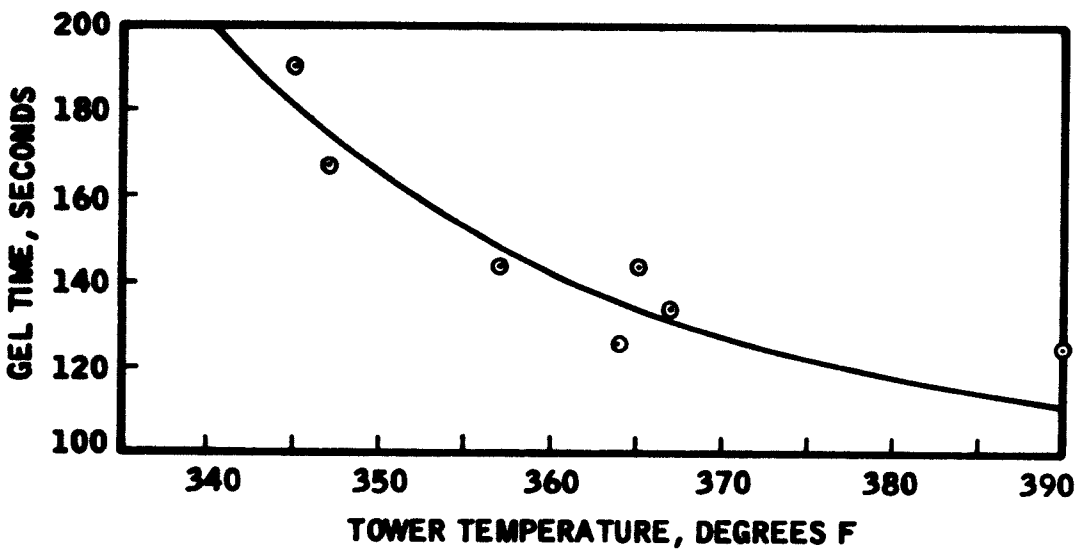


EFFECT OF RUNNING SPEED ON VISCOSITY

Figure 24

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

PROCESS CONDITIONS: RUNNING SPEED AT 65 FT/MIN.
RESIN SPECIFIC GRAVITY OF 0.853 AT 75°F

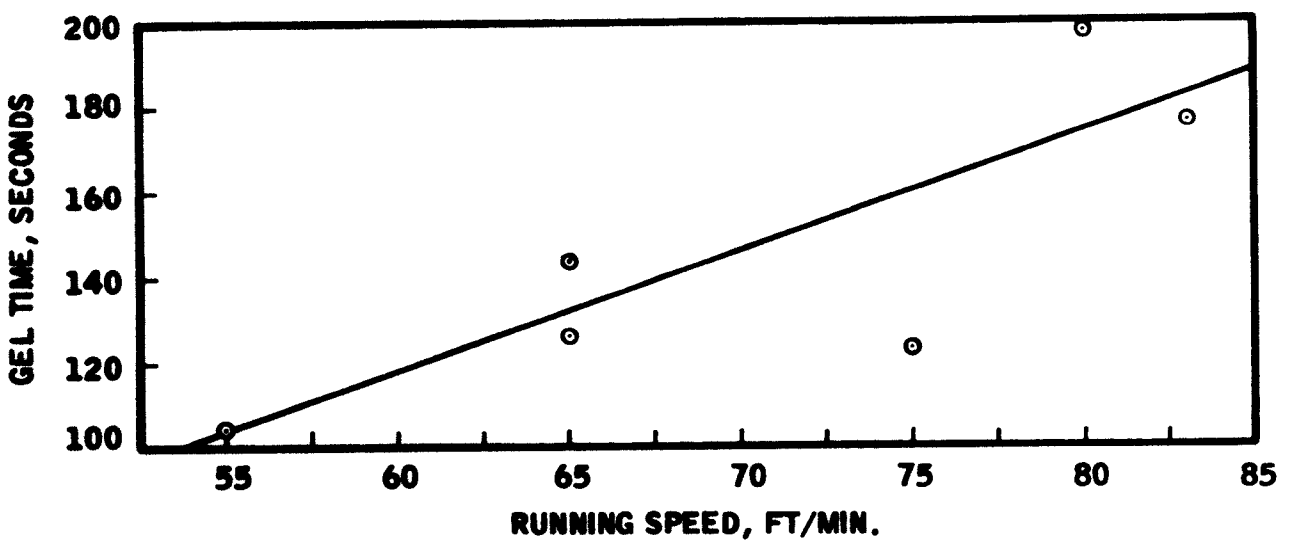


EFFECT OF TOWER TEMPERATURE ON GEL TIME

Figure 25

MATERIAL: 20 ENDS ECG 140'S HTS
58-68R RESIN SYSTEM, 0.55 PHR BDMA

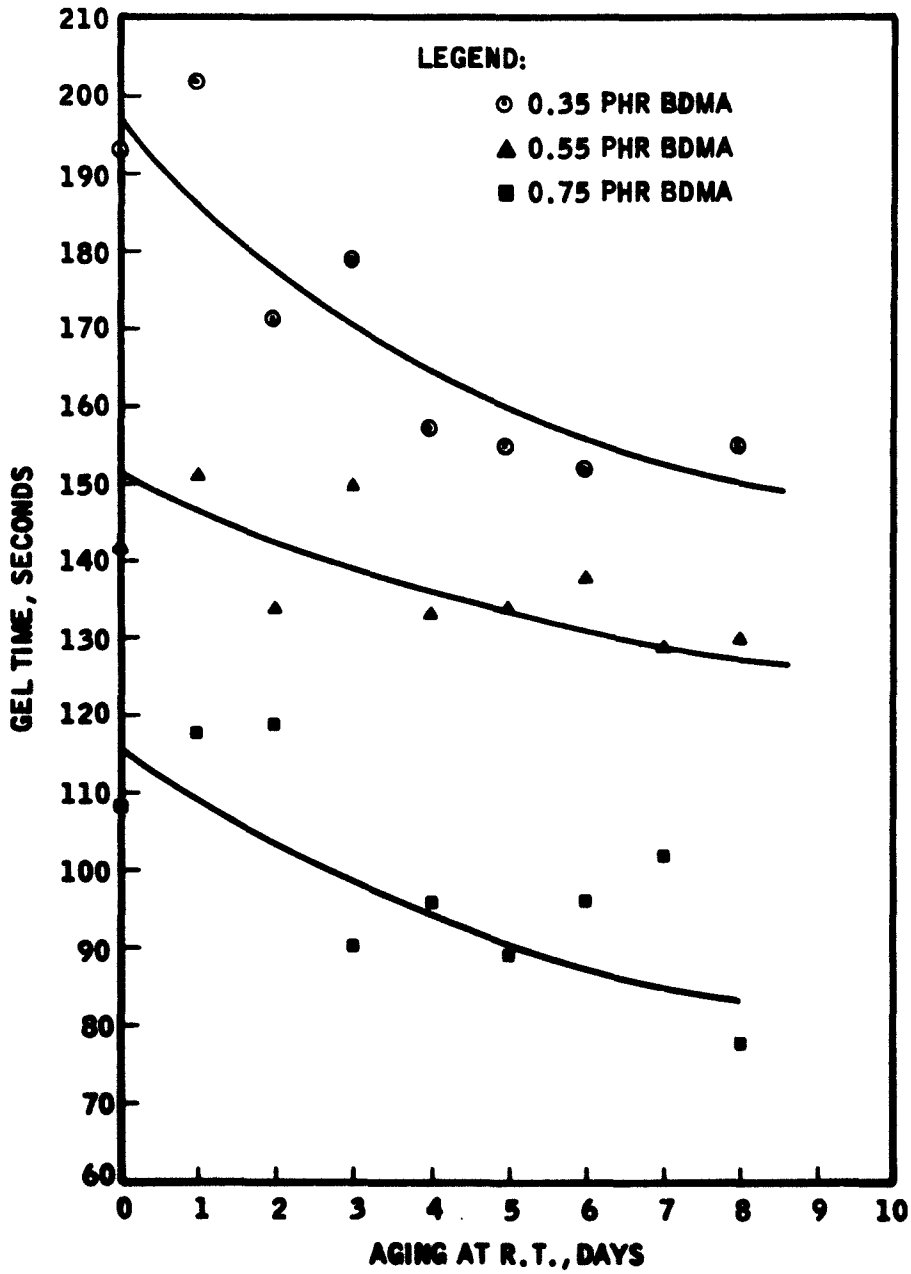
PROCESS CONDITIONS: TOWER TEMPERATURE AT 365° F
RESIN SPECIFIC GRAVITY OF 0.853 AT 75° F



EFFECT OF RUNNING SPEED ON GEL TIME

A270:63-899

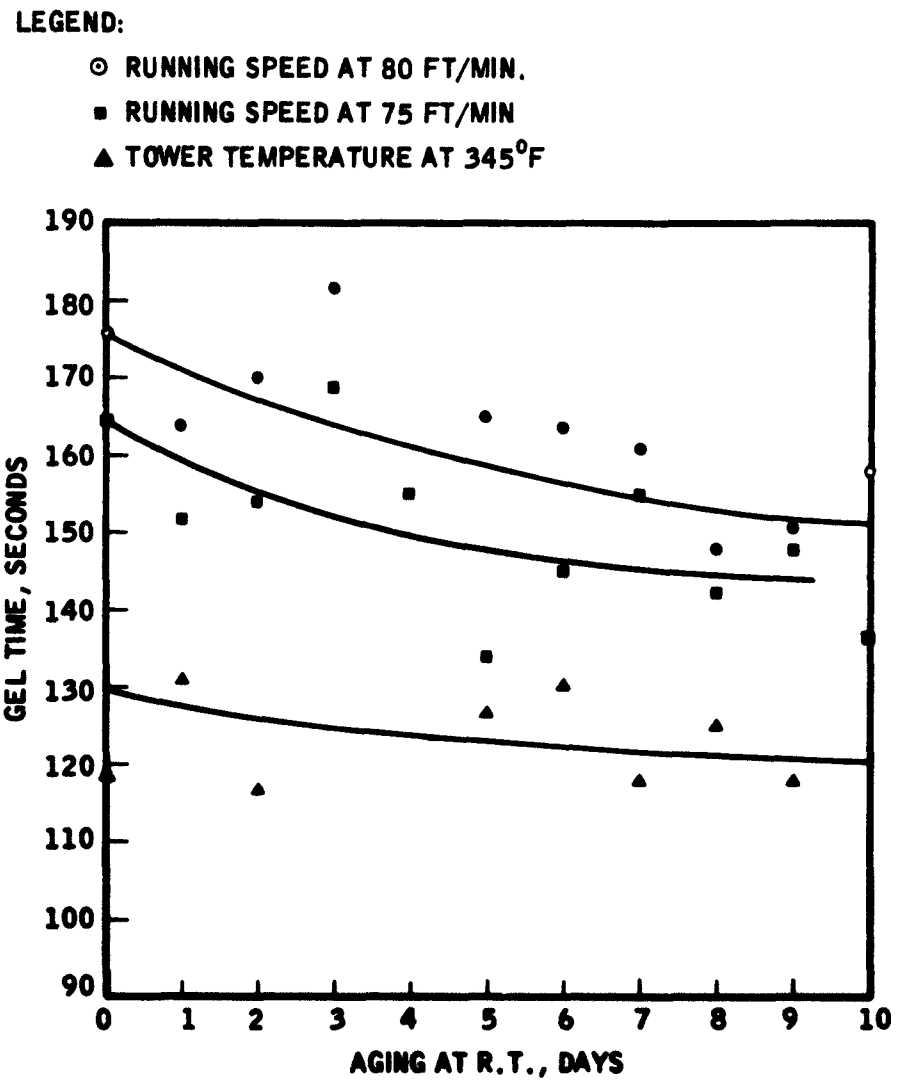
Figure 26



EFFECT OF AGING ON GEL TIME OF PREPREGS CONTAINING VARIOUS AMOUNTS OF BDMA

A270:63-897

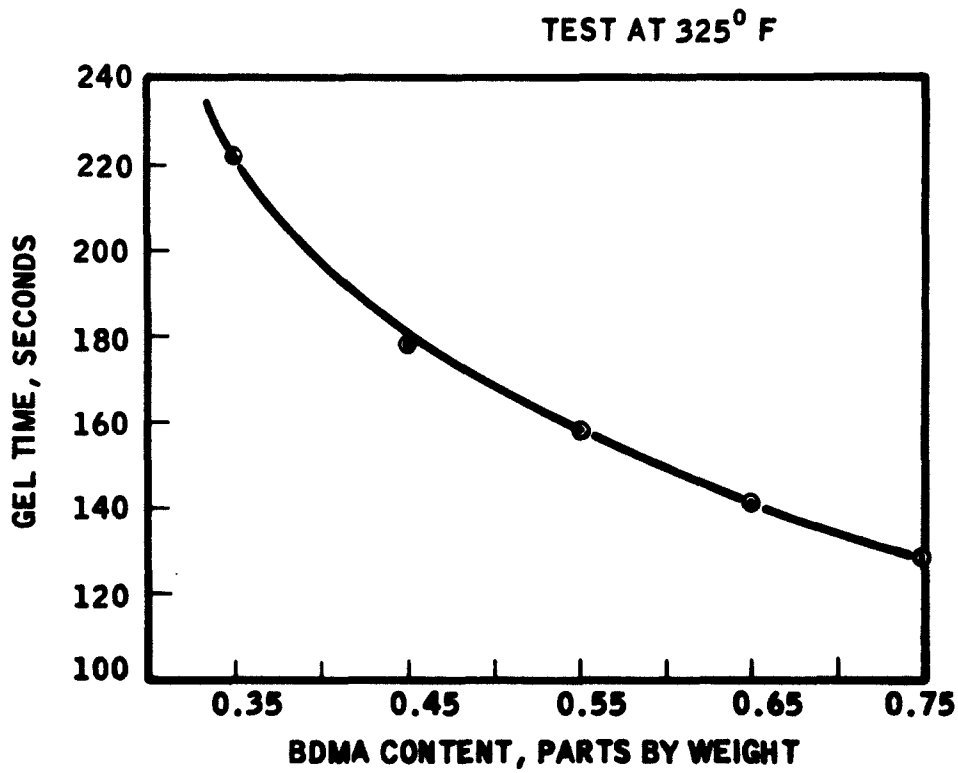
Figure 27



EFFECT OF AGING ON GEL TIME FOR PREPREGS PRODUCED UNDER VARIOUS PROCESSING CONDITIONS

A270:63-897

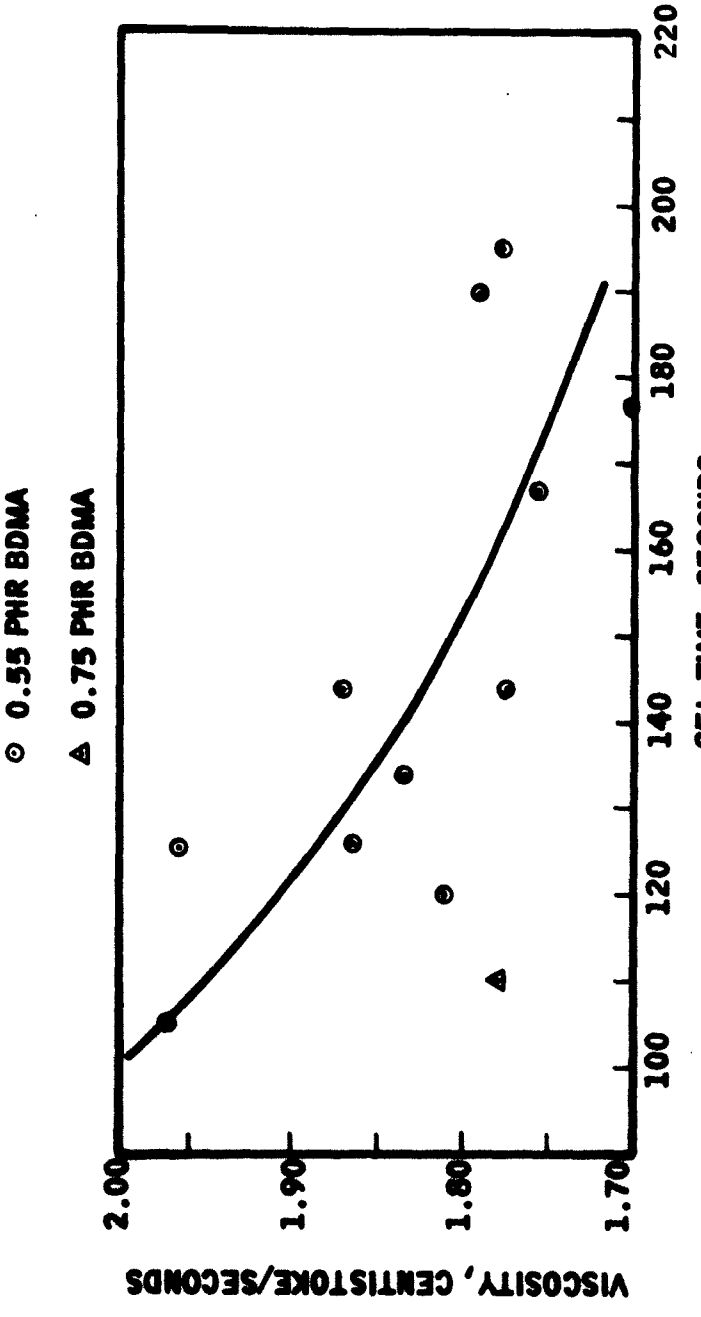
Figure 28



GEL TIME OF 58-68R RESIN SYSTEM WITH VARIOUS AMOUNTS OF BDMA

A270:63-900

Figure 29



CORRELATION OF GEL TIME AND VISCOSITY

A270:63-902

Figure 30

APPENDIX A

PROCEDURE FOR THE DETERMINATION OF VISCOSITY

The equipment for the determination of kinematic viscosity includes a refractometer, calibrated Type No. 100 Canon-Fenske viscometer, graduated cylinders, constant-temperature water bath, and a stop watch. The resin from 50 to 60 yards of prepreg material is extracted with approximately 50 ml of DMF (dimethyl formamide; refractive index of 1.4300 ± 0.0005) and the solution filtered through Grade 512 filter paper. A 15-ml quantity of the filtrate is then standardized with DMF to refractive index of 1.4480 ± 0.0002 (19.4% solution by weight of resin). For standardization purposes, 1 ml of DMF reduces approximately 0.0014 unit refractive index. A 12-ml quantity of the standardized solution is transfered to the Canon-Fenske viscometer, and the temperature of the solution stabilized at 25° ± 0.2°C in a constant-temperature water bath. (Passing the solution 3 times through the viscometer is normally satisfactory). Using a stop watch, time required to flow measured quantity of solution through the capillary tube of the viscometer is determined. An average efflux time is determined on three measurements taken for each solution with a maximum spread in measurements of 0.5 sec for the three readings. Efflux time is converted to viscosity in centistokes/sec by multiplying the time by a constant factor of the calibrated viscometer.

APPENDIX B

PROCEDURE FOR THE DETERMINATION OF GEL TIME

The equipment for the determination of gel time consists of the Fisher-Johns melting-point apparatus, glass cover discs, and a stop watch. A small segment of prepreg roving, approximately 1/8-in. long, is cut and placed between the glass cover discs. This specimen is placed on the melting-point apparatus, which is preheated to a constant temperature of 325°F (163°C). While heating, the resin is probed until gelation has occurred as observed through a magnifying glass. The gelatin or the end-point is reached when the resin viscosity is increased to a point where resin adheres to the glass surface and forms a mass of globules as contrasted to a clear, transparent liquid for the ungelled specimen. The gel time is that period of time between the placement of a specimen on the heated plate and the formation of a resin gel.

APPENDIX C

PROCEDURE FOR THE BAND WIDTH MEASUREMENT

For band width measurement with no pressure applied, prepreg roving unspooled directly from a package is laid flat on a table and measurements taken to the nearest 0.001 in. using a micrometer caliper. For band width measurement with pressure applied, a prepreg roving 6-ft long is tied at one end to a fixed pin or a holder, and a 10-lb weight is attached to the other end. The weighted end is then suspended over the edge of the table, which is measured 4 ft from the pin. Three band width measurements are taken along the length of the specimen at 1-ft increments from the pin. Areas of sudden band width change, such as those occurring at the traverse termination point of a waywind package, are avoided for the measurement. Another measurement is taken at the table edge, where the largest spread in band width occurs under tension.

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